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Manufacturing Methods and Technology Engineering

for

"Growth of Large Diameter Nd:YAG Laser Crystals"

Final Technical Report
October 1, 1979 to March 31, 1983
Contract No. DAAB 07-77-C-0375
Modifications P0003/P0004

by

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REPORT DOCUMENTATION PAGE

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1 REPORT NUMBER	2 GOVT ACCESSION NO
3 RECIPIENT'S CATALOG NUMBER	
4 TITLE (and Subtitle) Manufacturing Methods and Technology Engineering for "Growth of Large Diameter Nd:YAG Laser Crystals."	5 TYPE OF REPORT & PERIOD COVERED Final Technical Report Oct.1,1979-Mar 31, 1983
	6 PERFORMING ORG REPORT NUMBER
7 AUTHOR(s) Robert Uhrin and Roger Belt	8 CONTRACT OR GRANT NUMBER(s) DAAB 07-77-C-0375 Mod P0003/P0004
9 PERFORMING ORGANIZATION NAME AND ADDRESS Airtron Division Litton Industries, Inc 200 E. Hanover Ave., Morris Plains,NJ 07950	10 PROGRAM ELEMENT PROJECT, TASK AREA & WORK UNIT NUMBERS 2779751 DO-A7
11 CONTROLLING OFFICE NAME AND ADDRESS Department of the Army Fort Monmouth Contracts Office USAERADCOM,DRDEL-AQ-M-2 Fort Monmouth, N.J. 07703	12 REPORT DATE July, 1983
	13 NUMBER OF PAGES xi + 133
14 MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office) U.S. Army Night Vision and Electro-optics Laboratory Fort Belvoir, VA 22060 Attn: Dr. A. Pinto, DELNV-L-B	15 SECURITY CLASS (of this report) Unclassified
	15a DECLASSIFICATION/DOWNGRADING SCHEDULE

16 DISTRIBUTION STATEMENT (of this Report)

Approved for public release, distribution unlimited.

17 DISTRIBUTION STATEMENT (of the abstract entered in Block 20 if different from Report)

None

18 SUPPLEMENTARY NOTES

None

19 KEY WORDS (Continue on reverse side if necessary and identify by block number)

Laser Crystals
Nd:YAG
Yttrium Aluminum Garnet
Czochralski Growth

20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report describes Czochralski growth experiments on a production process for Nd:YAG boules. At program start most production techniques gave a boule about 1.25 - 1.50 inch diameter and 5 - 7 inches long. A goal of 2 inches in diameter, with similar length and quality, would nearly double laser rod yield for many Army programs. All experiments were performed on standard growth stations in a production facility.

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Power was supplied through a 450 kHz, 120 kVA, RF generator to heat a 4.5 x 4.5 inch cylindrical iridium crucible. Melt charges of 4.3 kg were employed which contained 1.1 atomic percent of Nd. The first few runs presented difficulties in enlarging the seed crystal to the finished diameter. Most boules were strained and cracked spontaneously. A technique of gradual enlargement of the seed worked more satisfactorily and allowed short sections of two inch boules to be grown. Cracking of boules was related to blossom formation after seeding and was connected with large radial gradients in the melt. Measurements were made to define and minimize these gradients for increased chances of large boule growth. Axial and radial gradients received more attention. It was found that the use of a Dy_2O_3 stabilized zirconia for insulation gave a very favorable growth gradient for our 4.5 x 4.5 inch crucibles. Twelve runs were started over a year. The first five of these were poor quality due to growth or apparatus difficulties. The next runs showed steady improvements which culminated in several good boules close to 2 inch diameter. These had few cracks, blossoms, or other defects. The first delivery of 12 engineering laser rods was taken from one completed boule. The tested rods were found to meet all specifications for the program. At least one boule was obtained which yielded more than 30 rods. Steady progress was made in growth procedures for the next several runs. The confirmatory sample gave 37 laser rods which met all specifications for AN/GVS-5 type rods. Boules were then grown for a pilot production run. The first attempt gave a very good boule which yielded 50 rods. All of these met specifications and objectives of the program. It appears that even larger boules are feasible with the proper growth equipment. Submitted test reports were accepted. A final demonstration of the growth process was made to industrial and military specifications.

Manufacturing Methods and Technology Engineering

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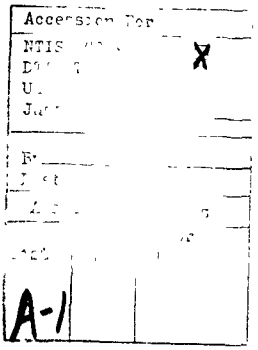
"Growth of Large Diameter Nd:YAG Laser Crystals"



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The objective of this program is to investigate methods for increasing the boule diameter of Nd:YAG production growth runs without deterioration of laser rod quality. A suggested goal of two inches would nearly double present rod yields. This goal was attained under the program to meet all AN/GVS-5 rod specifications.

PURPOSE

The reproducible growth of Nd:YAG for laser rods was developed in the late 1960's. It is performed exclusively now by the Czochralski method. All production boules are currently 1.25 - 1.50 inches in diameter. The purpose of this program was to obtain a larger yield of high quality rods by increasing the diameter of the grown boule. Preliminary investigation has shown that a goal of 2.0 inches is attainable and could nearly double rod yields for many Army requirements.

This program con of several parts including crystal growth, rod fabrication, and passive testing for quality. Laser rods provided under the program were required to meet existing military specifications for AN/GVS-5 applications. The major goal was achieved for a process which yielded a crystal boule which was 50 mm in diameter and 75 - 100 mm long and gave at least 30 laser rods per section.

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FOREWORD

This final technical report on Manufacturing Methods and Technology Engineering for "Growth of Large Diameter Nd:YAG Laser Rods" was prepared by the Airtron Division of Litton Industries, Inc., Morris Plains, New Jersey 07950 under Contract No. DAAB 07-77-C-0375 for the Solid State and Injection Laser Team of the U.S. Army Electronics Research and Development Command, Night Vision and Electro-optics Laboratory, Fort Belvoir, Virginia 22060.

The program was initiated by Mr. John Strozyk. During the next two years, it was monitored by Mr. William Comeyne, Ms. Kay Chloupek, and Dr. Albert Pinto. The production of Nd:YAG for laser rods under this program is achieved by the Czochralski method. These boules are currently 1.25 - 1.50 inch in diameter. The purpose of this program was to obtain a larger yield of high quality laser rods by increasing the diameter of the grown boule. Preliminary investigation has shown that a suitable goal of 2.0 inches nearly doubled rod yield for sizes used in the AN/GVS-5 and other Army programs. The same growth equipment and procedures are utilized as in all previous production.

The production program was under the supervision of Dr. Roger F. Belt, Research Director. Mr. Robert Uhrin was Project Engineer and performed all growth experiments. Ms. Karen Grimes was growth technician. All rods were fabricated and coated at the Airtron plant under the direction of Mr. Steven Turner.

Section I - Technical Summary

1.0 Introduction

Large single crystals of neodymium doped yttrium aluminum garnet (Nd:YAG) are required for future military program applications of optically pumped solid state lasers. From its discovery in 1964 until the present time, the most expedient method of obtaining such crystals of laser quality has been by means of the Czochralski growth procedure. In current production practice, this method consists of seeding and pulling a crystal from a melt contained in an iridium crucible. The crucible is heated by means of kHz radio frequency induced currents. While the process is a good one it has remained virtually unchanged except for improvements in diameter control systems. Early work on Nd:YAG growth has been described in several publications.[1-3] The Defense Department through the U.S. Army has sponsored two previous production engineering programs connected with the timely growth[4] and laser rod fabrication[5] of Nd:YAG. These programs have determined the crystal size, boule yield, fabrication methods, and hence cost of laser rods. In general because boule size and yields are limited, the larger the rod size the higher the cost. Unfortunately this relationship is not linear and laser rods of a size greater than (7 x 75)mm still merit a premium price.

At the conclusion of the growth program[4] on Nd:YAG, a production process was developed which yielded boules of 30-38 mm in diameter. For nearly ten years this process has remained the same and few workers attempted any improvement. In the period 1976-77 an increased demand for laser rods engendered an examination of procedures to increase yields. A concurrent objective was the lowering or stabilization of growth costs during a period of high inflation. The principal contributions toward the cost of a laser rod are iridium, electrical power, materials, and labor. Thus any process which limits or eliminates any of these would be beneficial. During the natural evolution of material growth technology, the trend has been to grow larger crystals. A valid question has been asked often; why not grow larger boules of Nd:YAG?

The justification for larger boules of Nd:YAG is based on the need for greater yields of high quality material at lower costs. This can be accomplished theoretically by the following methods.

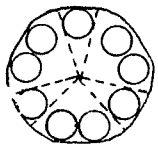
1. Grow crystals at current production diameters and maintain quality.

2. Grow larger diameter crystals at the same length and quality.

3. Improve the optical quality of boules by maintaining the melt composition fixed. This could occur in combination with any advance made in 1 and/or 2.

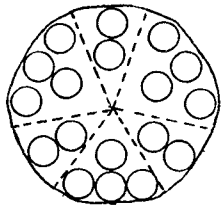
If objective 1 is chosen, the growth rate still remains fixed and not much is gained. This is further complicated by the Nd level in the crystal which constantly increases and eventually causes high strain or exceeds the laser rod concentration specification. If objective 3 is followed a substantial improvement results, but the time frame for realizing such an effort is certainly several years. Thus the most promising alternative is objective 2; to grow larger diameter crystals. An increase in diameter to about 50 mm would almost double the rod yield from a boule and seems to be within capabilities based on recent experiments.

Early experiments were begun at Airtron in 1976 and by 1978 a few 50 mm boules were grown with moderate success. These results led to an initiation of the present program to refine the technique for production purposes. The increase of boule diameter of any Czochralski grown crystal is a formidable task. Good methods have been developed for silicon, GGG, and sapphire over a period of years. The degree of difficulty is associated closely with the operating temperatures, number of chemical components, and factors which govern melt behavior. Nd:YAG growth is complicated by a melting point of 1975°C, a three component system, low distribution coefficient (0.18) for Nd, faceting phenomena, and high melt thermal convection. In addition the growth rate of Nd:YAG from the melt is a rather low 0.5 mm/hr. This places an extremely high demand on the temperature control system. Fluctuations of 10-20°C cannot be tolerated during the entire growth cycle of 2 - 3 weeks. At the present time there is no known method to increase growth rate without some sacrifice in quality. Hence for any planned increase of boule diameter, all the usual problems are not only present but also aggravated. In spite of inherent difficulties with Nd:YAG, it is safer to follow the Czochralski growth route rather than an entirely different procedure. In order to place our objectives of growth in perspective, let us recall that a popular size of laser rod required in large quantities is the (4.3 x 43) mm cylindrical type. A boule diameter increase from 35 mm at present to 50 mm will almost double the rod yield from a boule. Figure 1 shows a typical boule diameter at each chronological



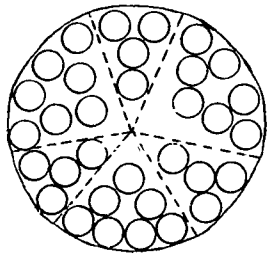
(a)

Initial Production
1967-1972
27 mm diameter; rods = 9



(b)

1972 PEM Results
1972-1978
38 mm diameter; rods = 18



(c)

Experimental
1979
50 mm diameter; rods = 33

Figure 1. Increase of Diameter and Rod Yield for Typical (4.3 x 43) mm Laser Rods

stage of growth development. It also records the number of laser rods of a (4.3 x 43) mm size that can be extracted from such a diameter. Notice that in Figure 1c an increase of only 5 - 6 mm in the diameter nearly doubles the rod yield from that of Figure 1b which is current production. Of course this increase has to be done at no sacrifice of quality or growth time. The following sections outline our experiments to solve persistent problems which are associated with increasing diameter.

2.0 Experimental

The basic approach utilized to achieve good quality growth has been to adjust the crucible position within the coil as a means of varying the radial temperature gradient in the melt. It is not clear what the correct gradient should be with the type of growth station design employed. However previous growth results indicate that a steeper radial gradient is required based on the severity of crystalline defects which have appeared prior to the crystal reaching final diameter. In all growth experiments the radial temperature gradient has been 25 - 30°C per centimeter. This is substantially lower than the existing gradient in production growth stations and has been a difficult parameter to control.

2.1 Growth Station Design

A conceptual view of the basic crystal growth station is presented in Figure 2. A 4.5 inch diameter and 4.5 inch high iridium crucible with cover (A) is supported by concentric zirconia tubes (B). This arrangement is surrounded by zirconia grain insulation (C) which is enclosed by a quartz glass tube (B). Power is applied to the crucible by means of an rf coil constructed from circular copper tubing (E). The area above the crucible into which the crystal is pulled is insulated by means of an alumina tube (F) and an alumina cover (G). Figure 2 shows the arrangement of these parts in construction.

2.2 Crucibles

In order to provide a situation for experimental growth similar to that existing in the production growth of smaller diameter crystals, the crucible size has been optimized at 4.5 inch diameter and 4.5 inch high. This insures that for a given length of crystal the neodymium dopant concentration typifies that of production crystals and permits the duplication of crystal growth rate. The charge for a

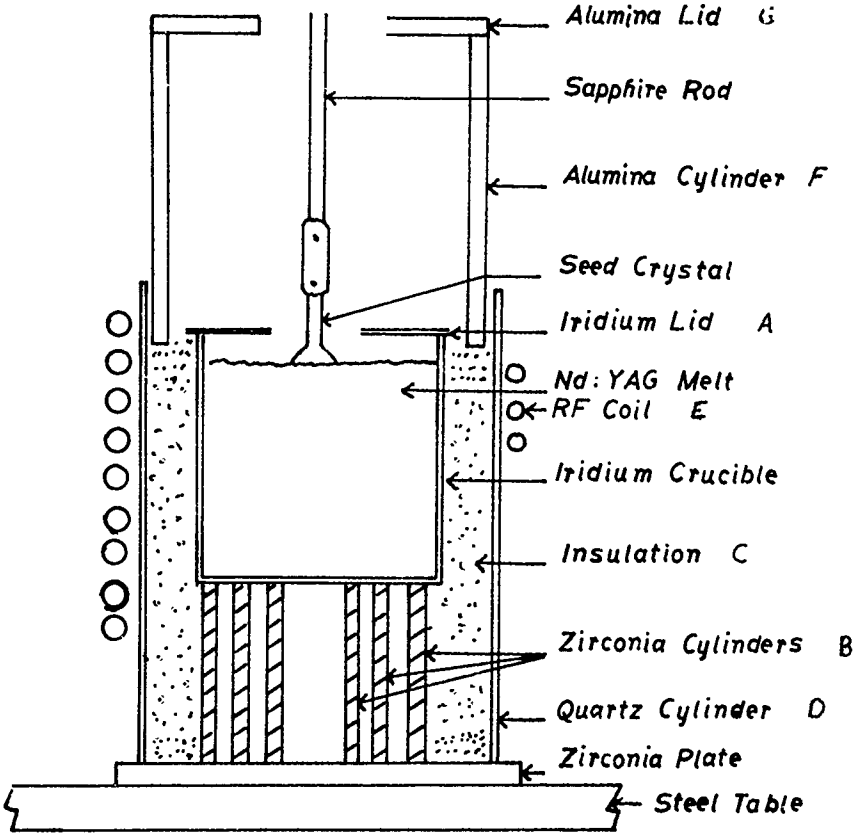


Figure 2 Drawing of Basic Growth Station

crucible of this size capacity is approximately 4300 grams. The expected weight of the pulled crystal is about 1 kg so no more than 20 - 25% of the melt is removed. Figure 3 illustrates the appearance of the crucible and its cover.

2.3 Raw Materials

Oxides used for experimental work are obtained from supplies used in the production growth area. These are readily available from commercial vendors at grades of 5-9's and 6-9's purity. In the case of the yttrium and neodymium oxides the purity refers only to the rare earth oxide assay, however. Thus care must be exercised to insure that contaminants do not affect the crystal growth or laser performance of fabricated rods.

2.4 Procedures

First the growth furnace is constructed by carefully aligning ceramic elements and the crucible for cylindrical symmetry. The oxides are blended to a homogeneous mixture and are then added to the crucible. In order to initiate growth, the melt temperature is adjusted to maintain the seed diameter when contact is made with the melt surface. If a diameter increase or decrease occurs, the temperature is adjusted upward or downward as required to maintain seed diameter. When pulling commences, the automatic diameter and temperature control systems are initiated and growth continues until the desired crystal length is obtained. The growth is then terminated and the entire furnace is cooled to room temperature over a period of several days.

Following completion of the growth run the crucible is cleaned thoroughly. This is accomplished by coring out the solidified melt with a diamond impregnated core drill. The crucible is then submerged in a container of molten lead fluoride which has an appreciable solubility for YAG. This treatment normally cleans the crucible to the point where a small amount of cleaning in acids readies it for use in the next growth cycle. Prior to this, however, it is tested for leaks and is repaired if necessary to prevent failure in the subsequent growth run.

3.0 Growth Run Results

Since the growth program was initiated continual progress has been observed in crystal quality. Quality is judged by

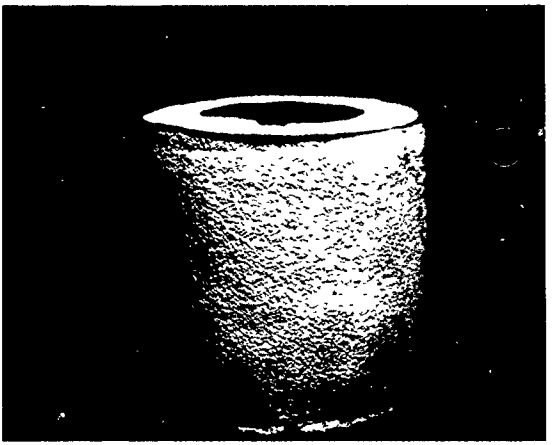


Figure 3 Iridium Crucible and Cover, 4.5 x 4.5 Inches

observation of a crystal in polarized and laser light. At the beginning all the crystals contained defects of a very gross nature.

3.1 Early Run Examples

Table I summarizes the growth runs completed during the course of the growth program. The first three growth attempts were designed to evaluate the performance of the control system with a new, larger power supply. The smaller 4 inch diameter crucible was utilized with a standard growth station design in order to make a comparison with results of growth runs made prior to initiation of the program. Some problems were experienced with melt contamination which originated from flaking of the quill used for holding the seed rod. This was caused by the higher temperature in the vicinity of the quill. Once the problem was identified the diameter control system functioned normally although poor control resulted from the effects of the contamination. For subsequent growth runs the larger 4.5 inch diameter crucible was put into service.

Growth runs beginning with run 2?.7 were performed with a water-cooled bell jar system. This approach was found to work very well since the excessive heat evolved from the growth furnace was effectively conducted away by the water-cooled enclosure. Without this system it would have been impossible to work in the vicinity of the growth furnace and the heat would have had a deleterious effect on the electronic control system.

3.2 Growth at Seed Diameter

Experience in the production growth of Nd:YAG indicates that the best results are obtained if the crystal is allowed to grow with a very steep solid/liquid interface projecting down into the melt. While this highly convex shape results in a core formation from facets developed at the tip of the growth interface, most of the strain is confined to a 3 - 4 mm diameter core region. High quality laser rods can then be extracted from the outer portion of the crystal cross-section and in between the radial strain lines.

An unfortunate consequence is that the disturbance of the crystal diameter normally results in a blossom (local high strain) fanning out from the central core. This situation also exists if the growth interface is not convex enough since

the faceted central region then has a tendency to trap liquid or secondary phases which result in defects.

Methods of insuring that the crystal maintains a highly convex profile are either to provide large temperature gradients or to utilize low rotation rates. The latter method alone is not very effective in the growth of Nd:YAG, since the rotation rate has little effect on interface shape except at high rotation rates (100 RPM). Thus the former method is resorted to for production growth.

For the situation which exists during growth of the larger diameter crystals care must be exercised, because the crucible size and growth station design tend to increase the temperature gradients. Efforts to increase the existing gradients can lead to cracking when the yield strength of the crystal is exceeded.

Most of the initial work during this program favored crystals which contain blossoms arising from a shallow interface shape. In many cases the resulting strain was so gross that extensive cracking of the crystals resulted. Blossom formation occurred at diameters of 0.5 - 1.0 inch. An alternate method of lengthening the growth interface was attempted and early results indicate that some improvement in growth occurred. In this instance initial growth was conducted at somewhat larger than seed diameter for an extended length and the crystal diameter was then increased slowly to its final value (Figure 4). It was felt that the additional heat sink capacity of the extended length of small diameter crystals would provide a steeper growth interface and therefore overcome the blossom formation in the 0.5 - 1.0 inch diameter range. Radiative losses could then maintain the steep interface as the crystal diameter was increased. Whether an improvement in growth results from this approach was not clear since only two growth runs were completed with this technique.

3.3 Measured Melt Gradients

It is felt that the most important parameter requiring control during the growth of Nd:YAG is the radial melt temperature gradient. Because of the crystal's high melting point (1950°C) it is difficult to measure a gradient directly by accurate methods. One approach which has been utilized satisfactorily is to scan the melt surface with the optical pyrometer used for diameter control. This method has been

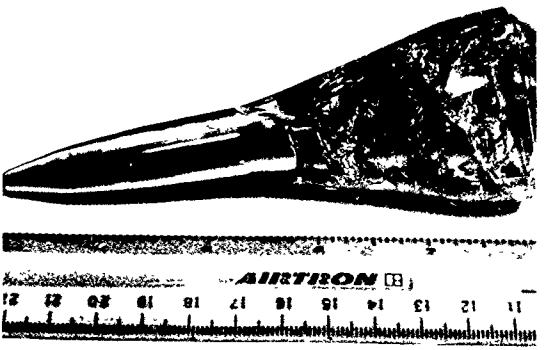


Figure 4 Growth From Slowly Increased Seed Diameter for 2 Inch Length

found to be repeatable and has proved to be useful for qualifying the growth results of various growth station designs.

Figure 5 illustrates the results of two such scans for different growth station designs. Although similar in some respects, these charts have one characteristic which may be related to blossom formation at small crystal diameters. It can be seen that the radial melt temperature gradient is higher at smaller melt radii and then decreases as the distance from the melt center increases. This means that constitutional supercooling can occur at small crystal diameter if the rate of the diameter increase exceeds the ability of the diameter control system to maintain the crystal on its program. Ideally the radial gradient should have a low slope more typical of that observed at the larger radii in Figure 5.

3.4 Review of Growth Runs

The bulk of the experimental work was reported in the interim reports but will be described briefly here. All growth runs are outlined in Table I. The initial growth runs (2141 - 2162) attempted to evaluate the performance of the power supply and growth station design with a smaller crucible. With introduction of a water cooled growth enclosure in run 2014 growth commenced in the large 4.5 inch crucible. Growth results were generally poor until run 2301. Attempts were made to obtain an extended length of small diameter growth which it was hoped would act as a heat sink and help to maintain a steeper growth interface in the low gradient environment. Somewhat better results were obtained but power supply failures prevented a yield of usable material in spite of flawless growth (2329).

An evaluation of the radial melt temperature gradient indicated that the available gradient was lower in the established growth station design than in production stations. Therefore it was felt that stable growth with the lower gradient could be obtained only at a lower growth rate. As a result, growth runs beginning with 2349 were made with a pull rate of 0.0125 inch per hour. This crystal was grown at a smaller diameter than normal, but the growth results appeared to support that conclusion. A power interruption toward the end of the growth run generated a blossom which led to cracking. However, the crystal was otherwise free of defects. No defects were present prior to the crystal reaching final diameter or shortly thereafter.

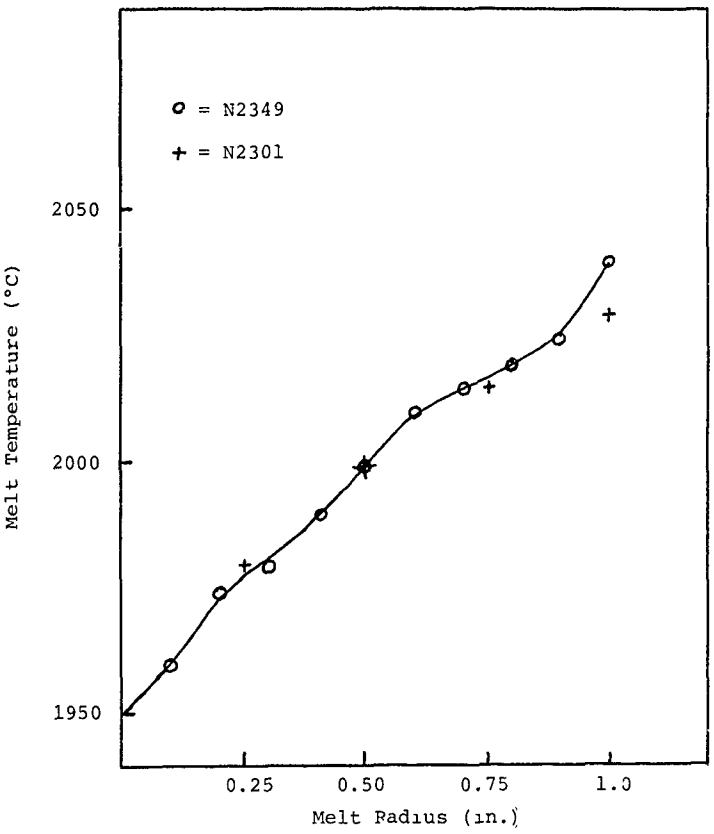


Figure 5 Nd:YAG Melt Temperature as a Function of Melt Radius

Table I

Summary of Crystal Growth Runs

Run #	Crystal Diameter	Pull Rate	Rotation Rate	Remarks
2141	1.40	0.020	15	No crystal. Run terminated due to melt contamination.
2154	1.40	0.020	15	No crystal. Run terminated due to melt contamination.
2162	1.40	0.020	15	No crystal. Run terminated due to melt contamination.
2014	1.75	0.020	15	Poor diameter control. Atmosphere control off due to bell jar leak.
2227	1.75	0.020	15	Three growth attempts with cracking. Final attempt with extended length at small diameter.
2301	1.75	0.020	15	Three growth attempts with cracking. Small blossoms in small diameter section.
2329	1.75	0.020	15	Two growth attempts. Crystal cracked when power failed No blossom.

Table I

Summary of Crystal Growth Runs

Run #	Crystal Diameter	Pull Rate	Rotation Rate	Remarks
2349	1.5	0.0125	15	Lower pull rate and smaller diameter produced flawless crystal which cracked from blossom generated by power interruption toward end of run.
2363	N/A	N/A	N/A	No crystal obtained in several growth attempts. Crucible was found to be leaking.
2429	N/A	N/A	N/A	No crystal obtained in several growth attempts. Crucible was found to be leaking.
2435	1.75	0.018	15	Crystal melted off after 1 inch of straight growth due to power supply problem. No apparent crystal defect but crystal cracked from melt off.
2463	1.85	0.0125	15	Crystal melted off after 2 inch of straight growth. Power supply problems led to power loss and crystal cracked. Only 2 small defects in straight section.
2464	1.95	0.0125	15	Power loss after 2 inches of straight growth. Crystal cracked during quench. Poor diameter control. Large blossom after reaching diameter.
2484	1.95	0.0125	15	Crystal melted off after 2 inch of straight growth. Good diameter control. Crystal had large blossoms first before reaching diameter.

Table I

Summary of Crystal Growth Runs (Continued)

Run #	Crystal Diameter	Pull Rate	Rotation Rate	Remarks
2500	1.90	0.0125	15	Good diameter control but crystal quality was poor. Crystal cracked during cooling. Run proceeded to full extent.
2533	1.95	0.0125	15	Good diameter control Crystal had large blossom before reaching diameter and small blossoms late in run. Crystal yielded engineering sample of 12 rods.
2542	1.90	0.0125	15	Good diameter control but crystal of poor quality. Run proceeded to full extent and crystal did not crack during cooling.
2572	1.90	0.0125	15	Good diameter control. Crystal flawless except for blossom at end of growth cycle.
2649	1.90	0.0125	15	Good diameter control. Crystal was flawless for full length of growth.
2627	1.90	0.0125	15	Several unsuccessful growth attempts due to power supply problems.
2682	N/A	N/A	N/A	Crucible leaked.
3048	1.90	0.0125	15	Power supply failed. Crystal cracked when pulled from melt. Extensive internal defects.
3079	1.90	0.0125	15	Three unsuccessful attempts. Crucible leaked in third attempt.

Table I

Summary of Crystal Growth Runs (Continued)

Run #	Crystal Diameter	Pull Rate	Rotation Rate	Remarks
3111	1.90	0.0125	7.5	Good diameter control. Small internal defect as crystal reached diameter.
3122	1.90	0.018	4	Crystal cracked on first growth attempt. Excellent control and quality on second attempt. Pilot production run.
3163	1.90	0.018	4	Attempt to duplicate prior growth run. Crystal cracked during cooling. Internal defects.

The next two growth runs were not completed because of leaks which developed in the crucible wall. However the following experiment (2435) was performed at a slightly larger diameter but at the customarily higher growth rate. This crystal melted off after a short section of growth at diameter due to a power supply problem. Since there were no evident crystalline defects prior to this occurrence this result would tend to dispute the lower growth rate argument. Nevertheless the next growth run (2463) was made at the lower growth rate and in spite of another power supply problem the crystal had only two small defects in the at-diameter section.

Beginning with run 2464 a significant improvement was observed in the crystal growth results. Although there were still problems with defect generation the general crystal quality improved to the point where extended lengths of growth at diameter were attained without cracking. Growth runs 2464 and 2484 virtually duplicated the previous experiment. In each case there was a large "blossom" generated as the crystals reached diameter and they cracked after reaching about two inches of growth at diameter. The following growth run (2500) was also of poor quality, but growth of this crystal proceeded to its full extent before the crystal cracked during the cooling process.

The subsequent four growth runs all proceeded to completion without cracking and the first sample of laser rods was extracted from crystal 2533 (Figure 6). This crystal contained defects on each side of the section from which the laser rods were obtained, but this central section was of good quality. Although the following growth run (2542) was of poor quality, the next two growth runs (2572 and 2649) were of excellent quality and these crystals yielded the required laser rods for the demonstration samples. These crystals are shown in Figures 7a and 7b.

The final group of seven growth runs generally attempted to continue the procedures established in the earlier growth experiments. Difficulties encountered during these runs revolved mainly around the reliability of the power supply and crucibles. With the exception of runs 3048 and 3163 (internal defects) and 2682 (leaked before growth commenced), the overall crystal quality was found to be good prior to premature termination of the growth. In the case of cracking during the growth cycle, failure was probably due only to thermal stresses (i.e. not defect related) and can be corrected by suitable modification of the growth station design. This was particularly evident in crystal 3122 which

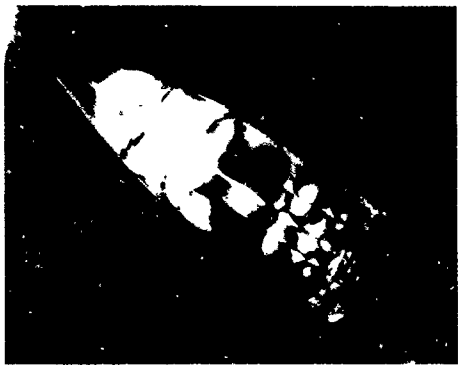


Figure 6 View of Boule N2533 Between Crossed Polarizers

Figure 7(a) Boule from Run N2572. Ordinary Light A yield of 37 rods was obtained.

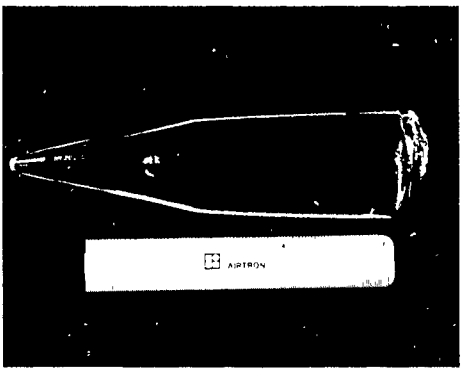


Figure 7(b) Boule from Run N2649. Ordinary Light.

cracked on the initial attempt but contained no defects after cooling. Upon remelting and regrowth an excellent quality crystal was obtained. This pilot run yielded 62 laser rods, 30 of which were processed for the sample. Figures 8a and 8b show the crystal in ordinary light as well as longitudinally between crossed polars.

The interesting note is that whereas a large portion of the growth runs were conducted at a slow growth rate the eventual success was realized at the faster pull rate of 0.018 in./hr. This is believed to have resulted from the improved growth station design with incorporation of the Dy_2O_3-ZrO_2 insulation as well as experience gained in working with the large system.

3.5 Problem Analysis

The major difficulty which has prevented growth of larger diameter crystals of high quality has been the internal blossom generation at small crystal diameter. This has led to an inordinate amount of strain in most cases and finally the crystal cracks. However, even in the cases where the strain from blossom generation is comparable to that encountered in production growth of smaller diameter crystals, cracking has occurred. It would appear, therefore, that the larger diameter crystals are unable to withstand the greater differential thermal stress between the cold surface and hot center of the crystal. A simple remedy for this appears to be a top heater.

The most recent growth results indicated that defect-free crystals of even larger diameter should be obtainable. While improving the growth station design, attention should be given to the area above the crucible into which the crystal is pulled during growth. Better insulation of this chamber should reduce the thermal loss and thus the differential thermal stress and tendency toward cracking. However, this is expected simultaneously to affect the radial and axial gradients so that blossom generation is prevented and thermal shock is alleviated.

The greatest difficulty in this program however, has come from defect generation as the crystal approaches final diameter in its growth cycle. Although constitutional supercooling is inherent in the growth of Nd:YAG and slow growth rates must be utilized as a result, the aforementioned problem does not seem to be related mainly to growth rate.

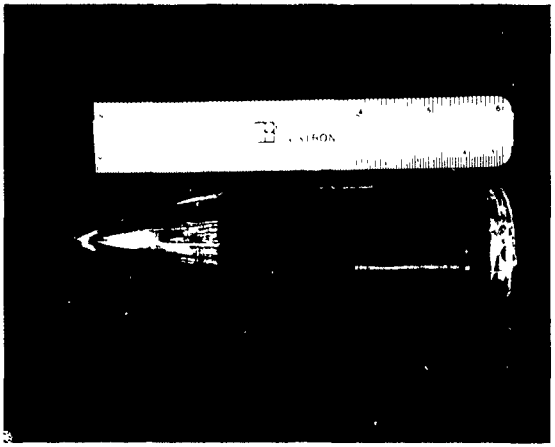


Figure 8 (a) Crystal N3122 Viewed in Ordinary Light.

Figure 8(b) Crystal N3122 Viewed Longitudinally Between Crossed Polarizers

Data on the typical radial melt temperature gradient obtained with the growth station design used for this work was presented earlier in the report. The most significant aspect of these data is that the gradient does not change much with small changes in the growth station design. While it would be desirable to obtain a gradient as near as possible to that existing in production, this leads to some serious problems with the growth stations involved.

One important feature is that a gradient of the required magnitude would push the crucible wall temperature close to the iridium melting point. This would make operation at the growth temperature marginal and associated growth operations procedurally difficult. Another feature is that the additional heat loss from the growth station due to a higher radial gradient puts some constraints on the power supply in that the higher power output required raises the output voltage to a point where high voltage discharges tax the power supply's reliability. In spite of these difficulties a unique solution was found to modifying the radial melt temperature gradient. There appears to be a direct association between the institution of this change and the better growth results toward the end of the above experimental work.

It should be noted that most of the insulating ceramics utilized for crystal growth are composed of zirconia. While this material possesses good thermal insulating properties by virtue of its relatively low thermal conductivity, it is also quite transparent to blackbody radiation at the crystal growth temperature.

Figure 9 represents a curve of the blackbody radiation from an emitter radiating at 2300°K, the approximate melting temperature of Nd:YAG. The peak of this curve lies at about 0.7 μm where zirconia is transparent. Thus much of the infrared energy passes through the normal insulation used in the growth station. A material doped with Dy^{3+} has very strong absorption at this same wavelength. (See Figure 10) A novel approach was utilized in order to effectively limit the amount of radiation escaping from the growth station and thus improve the insulating properties.

An alternate type of zirconia insulation was prepared by crystallizing the cubic form of ZrO_2 stabilized with Dy_2O_3 rather than Y_2O_3 or CaO which are normally used. This material was prepared by growing Dy_2O_3 (40 mole percent) stabilized cubic zirconia crystals with a patented

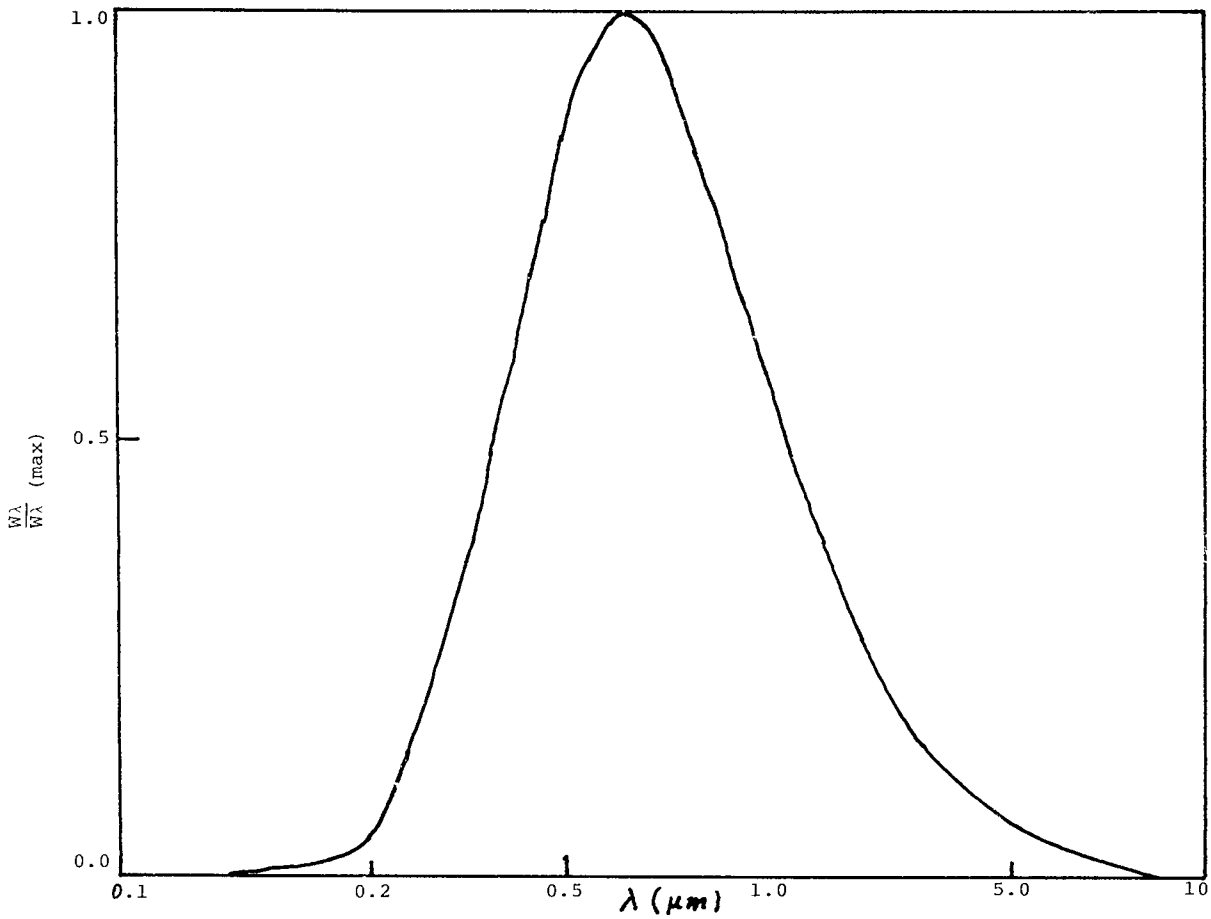


Figure 9 Emission from a Blackbody at 2300K

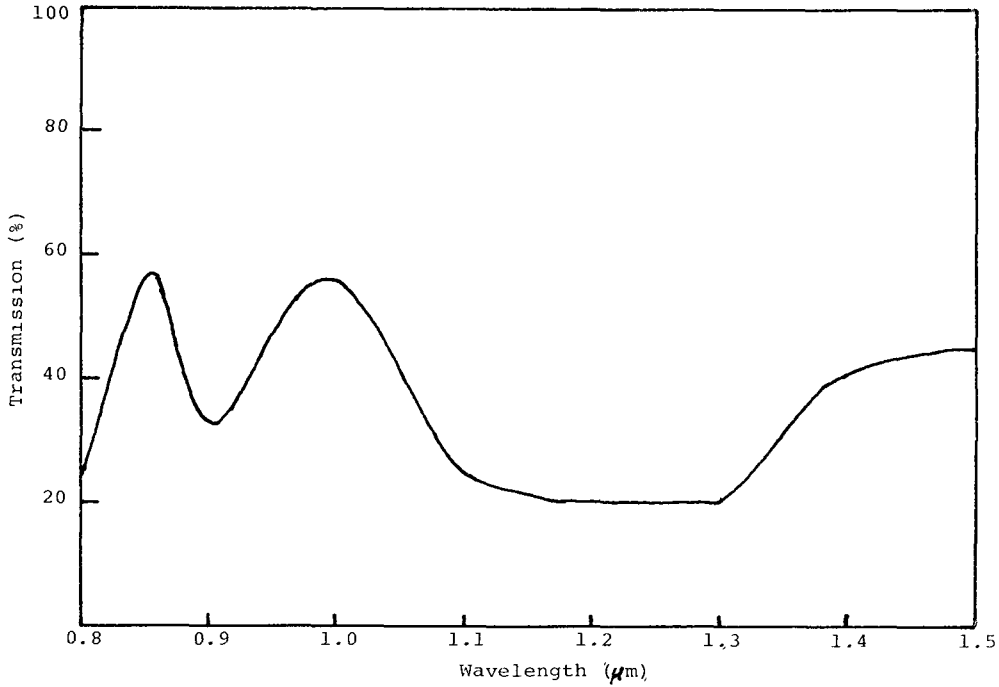


Figure 10 Absorption of Dy_2O_3 Stabilized Zirconia

growth process and then reducing these crystals to a granular form compatible with the growth station design. This procedure was initiated with growth run N2533 where the top two inches of insulation surrounding the crucible were replaced with the alternate insulation. A pyrometric probe of the radial melt temperature gradient for this run showed what appeared to be a refinement of the gradient near the center of the melt. While this was a gross measurement the true effect was realized when this crystal growth cycle was brought to completion without the crystal cracking. This was the first time a growth run at the large diameter was brought to completion in such a manner. The three subsequent runs also were completed by replacing all of the insulation surrounding the crucible with this alternate material. The absence of cracking in spite of extensive flaws (N-2542) indicated lower bulk crystal strain using this approach.

The real effect of this design change is not completely understood at this point but it is theorized that instability in the melt convection has been eliminated near the melt top center. Thus the tendency for generation of defects at the crystal core has been reduced. The next problem which has to be dealt with is some further modification of the growth interface to offset the tendency for defect generation due to constitutional supercooling. This has been approached initially by reducing the growth rate. However, this is an undesirable situation for improving the growth efficiency. It is believed at this time that the thermal convection in the absence of crystal growth is quite similar to standard production crystal growth. Further refinement in the growth can be expected, therefore, by evaluating the effects of crystal rotation rates on the growth quality.

4.0 Conclusions

It was demonstrated early in our program that large Nd:YAG melts can be handled successfully for growth of 2.0 inch diameter boules by means of the Czochralski method. Ideally the crucible diameter should be about twice the boule diameter. The 50 kW and 450 kHz production radio frequency growth stations can melt easily the charges of 4.3 kg needed for large boules.

In order to grow good quality crystals, careful control of both longitudinal and radial melt gradients is necessary when a steep interface is present and faceting occurs. The

most important of these is the radial gradient and for a large system it can be reduced by a judicious choice of insulation. Zirconia with a stabilizing additive of Dy_2O_3 was found to give good results.

With an optimized growth station geometry and a given growth rate of 0.5 mm/hr, the remaining variable is rotation rate. In order to match the melt isotherms closely to the growth interface, values of around 15 rpm or less gave a high quality boule free of precipitates or strain.

A growth process was developed which gave finished boules meeting the suggested goals of 50 mm diameter and 75 - 100 mm long. Late in the contract, boules were obtained which yielded 40 - 60 laser rods of a (4.3 x 43) mm size. This was well above the goal of at least 30 rods which completely meet a current AN/GVS-5 specification.

All laser rods were fabricated by a batch process developed for polishing 15 rods in a single fixture. The engineering, confirmatory, and pilot production samples were extracted from production boules and fabricated under an existing rod process. Quality control passive tests of the rods showed that specifications were retained by more than 90% of the extracted rods.

It appears that the boule growth results obtained under this program are transferable to current production stations. Since only one station was in operation the entire length of the effort, insufficient growth statistics were generated to forecast high boule yields. Thus some problems of cracking, blossoms, poor starts, equipment failures, and materials choice are still apparent. On the average these are no worse than results obtained with smaller diameter boules.

Results obtained here indicate that further increases in boule size are possible. However the proper RF unit, diameter control, and furnace geometry need to be combined for growth near 3.0 inches.

Section II

Process Specifications for Boule Growth and Rod Fabrication

1.0 Introduction

This section presents in detail the best developed process for growth of 50 mm diameter Nd:YAG boules. It is assumed that growth equipment of some type is available. This may consist of 400 kHz radio frequency units, 20 - 30 kHz solid state generators, or even low frequency motor generators. Each of these must be of 60 kW or larger power output to obtain stable melts of about 4.5 kg in iridium crucibles. Furthermore it is necessary to provide some type of automated diameter control system. This may be of an optical or weight sensing type with appropriate proportional power control or feedback to the generator.

After boules of Nd:YAG are obtained from the growth process, the rod selection and fabrication process can begin. Under normal manufacturing operations, a mixture of many rod sizes is desired. However for the objectives of this program, only one size rod is necessary. Thus the boule length is controlled at 90 - 100 mm in order to obtain two possible tiers of rods each of whose length is 43 mm. In this procedure the yield of rods is maximized from existing material.

The rod manufacturing process was perfected at Airtron nearly 15 years ago. It began by blocking each rod and polishing them one at a time. In the mid 1970's as laser rod volume increased, techniques were examined for fabricating rods in blocks of 10 - 30 where possible. The benefits of this multiple rod fabrication are favorable to large quantities of a single rod size. Thus all rods for this program utilized procedures which were designed for production rate of 100 - 300 per month. These rates assume that the requisite number of boules are provided as input.

2.0 Boule Growth

Growth of Nd:YAG, independent of crystal diameter, follows that of other oxide crystals familiar to the community. This includes laser hosts as well as optical crystals. Consequently, the combination of materials, components and processes should seem quite familiar. Some aspects outlined here are not intended to be construed as required for the

successful production of two-inch diameter crystals. In some cases individual preferences may be to expand certain facets of the process or to substitute materials or components where these have been shown to be interchangeable.

2.1 Materials and Components

The following Table II summarizes materials components and the sources for growth of the large diameter crystals.

2.2 Growth Station

A general drawing of the growth station was presented in Figure 2. Specific dimensioning for the components is provided in Table III. A photograph is given in Figure 11. A close view of the crucible is given in Figure 12.

2.3 Growth Process

The initial portion of the growth process consists of cutting all ceramic elements to the proper size prior to arrangement of the growth furnace. These are then dried in an oven to eliminate moisture introducted during the sawing process. This having been done the growth station is constructed.

All components are centered with respect to the centerline of the pulling mechanism. The zirconia cylinders are placed concentrically within the outer quartz sleeve. Zirconia grog is then packed within all open areas to the top of the zirconia cylinders. This having been accomplished the iridium crucible (Figure 13) is wrapped with a layer of zirconia felt which is secured to the crucible with sewing thread. The crucible is then centered atop the zirconia cylinders. The specially prepared Dy_2O_3-ZrO_2 insulation is added around the crucible to a position just below the top and the crucible lid is placed atop the crucible. Care should be exercised to insure that no contamination occurs to the crucible's internal surface. The growth station is completed with the addition of the alumina cylinder and lid which act as an afterheater.

The growth furnace having been arranged, it is surrounded by a pyrex bell jar and the system is purged with a mixture of nitrogen with 0.05% oxygen. Power is also supplied at a low level to the crucible to eliminate residual

Table II

Material Specifications for Nd:YAG Growth

Material	Application	Specification	Approved Source
Y_2O_3	Growth	5-9 purity (min)	Cerametals, Inc. Molycorp NIAC Research Chemicals Rhone-Poulenc United Mineral & Chemical
Al_2O_3	Growth	99.98 purity (min)	Baikowski International Cerametals, Inc. Rubis Synthetique
Nd_2O_3	Growth	5-9 purity (min)	Molycorp NIAC Research Chemicals Rhone-Poulenc
ZrO_2 (ceramic)	Furnace Construction	99+ purity	Leco Corp Zircoa
ZrO_2 (grog)	Crucible Insulation	99+ purity	Zircoa
Zr_2O (felt)	Crucible Insulation	99+ purity	Zircar Products, Inc.
Al_2O_3 (ceramic)	Furnace Construction	99+ purity	Norton
Al_2O_3 (ceramic)	Seed Rod Support	99+ purity	McDanel Refractory & Porcelain

Table II (Continued)

Material Specifications for Nd:YAG Growth

Material	Application	Specification	Approved Source
SiO_2 (fused)	Furnace Construction	Std.	Amersil
Ir	Crucible	99.9+	Engelhard Industries Johnson Matthey
N_2	Growth Atmosphere	99.99	Approved Vendor
O_2	Growth Atmosphere	99.8	Approved Vendor

Table III

Component Dimensions for Nd:YAG Growth Station

Component	Dimension (In.)
Alumina Lid	1/2 thk x 3 I.D. x 6 O.D.
Alumina Rod	As required
Alumina Cylinder	1/2 thk x 6 O.D. x 8 high
Seed Crystal	As required
Iridium Lid	0.090 thk x 3 I.D. x 4 3/4 O.D.
RF Coil	7 1/2 I.D. x 5 3/4 high x 3/8 copper
Iridium Crucible	0.090 thk x 4 1/2 I.D. x 4 1/2 high
Insulation	As required (Dy_2O_3-ZrO_2 to bottom of crucible)
Zirconia Cylinder	1 1/2 O.D. x 4 3/4 high
	3 O.D. x 4 3/4 high
	4 O.D. x 4 3/4 high
Quartz Cylinder	7 O.D. x 9 1/2 high
Zirconia Plate	1/2 thk x 7 1/8 dia.

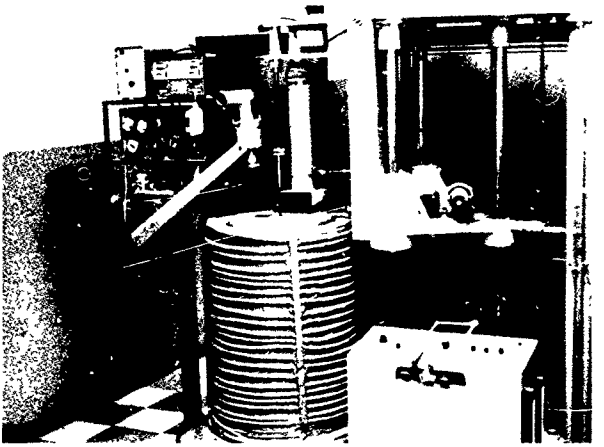


Figure 11 General View of Nd:YAG Growth Station

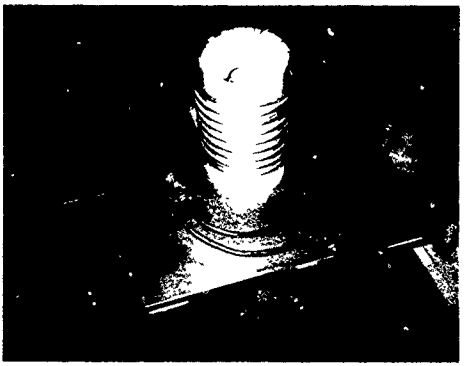


Figure 12(a) Basic Crucible and Bottom Insulation

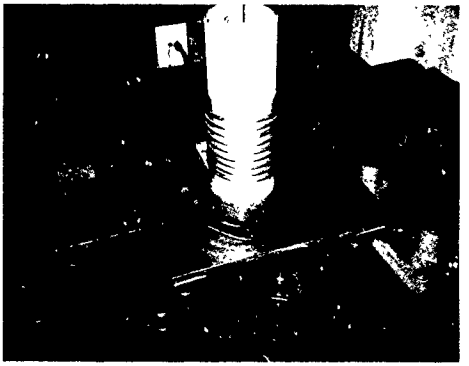


Figure 12(b) Top Alumina Tube and Cover Attached, Seed Rod Lowered

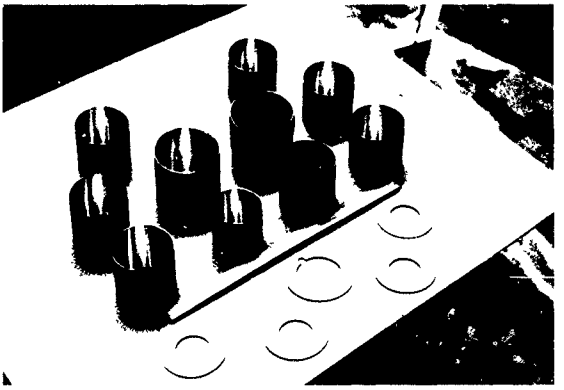


Figure 13 Group of Iridium Crucibles for Nd:YAG Growth

moisture within the growth station. While this is being done the crucible charge is prepared. The individual oxide components are weighed to 0.1 gm and blended for about one hour to insure thorough mixing. This mixture is then placed in clean beakers in preparation for loading of the crucible.

The crucible loading procedure should normally take several hours and is accomplished by gradually feeding the oxides into the crucible through a quartz tube while the power is increased slowly to melt the material. This operation is completed with a temperature at the melt center established below the crystal melting point. A seed and seed holding mechanism is then fed into the growth station in preparation for initiating growth.

Prior to actual growth a solid crystalline mass is generally present at the top center of the melt. This is dissipated by further adjustment of the power upward. A crystalline seed of the proper orientation is then dipped into the melt and the power is further adjusted to melt any solidified material back to the seed diameter. At that point the pulling operation is commenced.

In order to obtain the highest quality an automatic diameter control system is utilized. The crystal is smoothly programmed from seed diameter out to its desired diameter and growth is allowed to proceed until a satisfactory length is obtained. At that point the pull and rotation are stopped and the power is programmed down over several days.

Following extraction of the crystal, the crucible is removed from the growth station, the residual solidified melt is removed, and the crucible is cleaned. The growth station is then rearranged with a clean crucible and the growth process is repeated.

2.4 Boule Inspection

Having been removed from the crucible, the crystal is examined for extraction of laser rods. The initial step in this process consists of observing the strain pattern as the crystal is held between the crossed polars of a polariscope. This permits highly strained areas, particularly those originating from defects, to be identified. Once this has been accomplished the crystal is marked for usable length and end cut with a diamond blade. Following this procedure the end faces of the crystal are given an inspection polish and the crystal is again observed between crossed

polars but this time along its length. In this way more discrete areas of strain can be identified. If these are present the end face of the crystal is marked with a diamond scribe to delineate areas to be avoided during laser rod extraction.

The crystal is then given one final examination, this time to identify any light scattering centers within the bulk. This is accomplished by shining a high intensity light through the side while observing the crystal end-on. Careful scrutiny permits observation of individual scattering centers, the cross-sectional location of which can be marked on the crystal's end face.

The examination process having been completed, the crystal end face provides a cross-sectional map of unacceptable areas. One other marking scheme is used to designate high strain areas which exist in every crystal. The first of these consists of the highly strained central core area which arises due to the highly convex growth interface and extends for several millimeters in diameter at the very center of the cross-section. The second of these consists of six narrow radial areas projecting from the center out to the six facet lines running down the side of the crystal. Compositional inhomogeneities in these areas do not provide high quality laser rods so these areas must be avoided.

Having a complete map of the crystal's end face, it is now possible to choose locations suitable for extraction of laser rods. A template is used to designate possible laser rod locations dependent on laser rod diameter and length. These are similarly marked with a diamond scribe and recorded elsewhere for a permanent record. The crystal is then transferred to the fabrication area to complete the laser rod fabrication process. An example of two core drilled sections is given in Figure 14. The left boule is from a normal production run while the right is from a large diameter contract type boule.

3.0 Preparation of Laser Rods

3.1 Specification and Process

All laser rods delivered under this program were prepared to the specifications and drawing of Figure 15. This rod is identical to the AN/GVS-5 laser rod which is manufactured by several companies at the present time. The large quantities of the end item dictate that the rod cost be kept low. The method of manufacture of the range finder

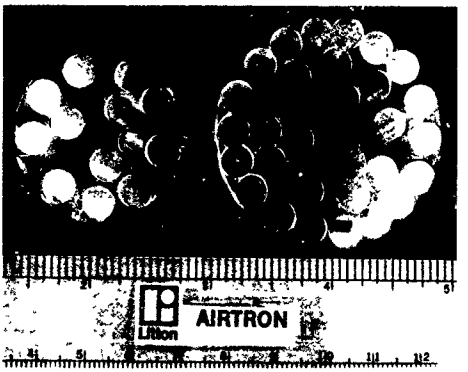
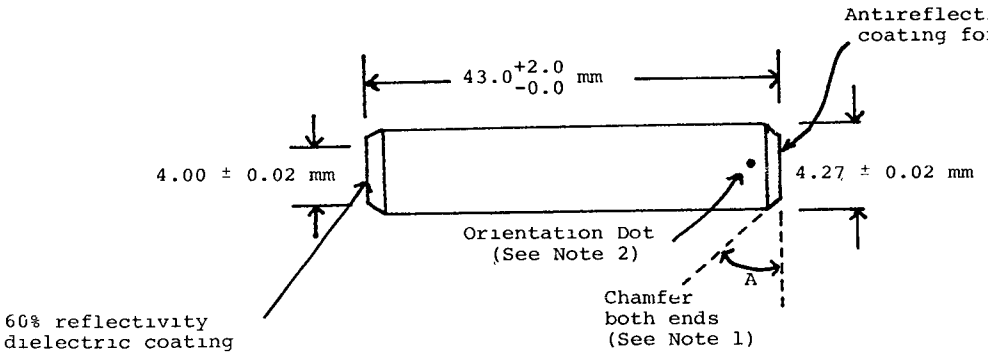


Figure 14 Core Drilled Nd:YAG Section; Left, Normal Production; Right, Large Diameter Contract Boule



Notes: 1. Chamfer surface to be rough finish, 20 to 30 microinches. Chamfer angle A = 45° ± 5°.
2. The antireflection (AR) coated end of each rod shall be identified with a dot or number located within 3 mm of rod end.

Figure 15 Dimensions and Tolerances of Laser Rod

also prevents reuse of the laser rod after installation. If the rod is abused, a complete refabrication becomes necessary. However this rarely happens in practice. If the rod passes all tests and specifications at the fabrication stage, the probability of not working in the final device is very low.

The boule material for the laser rods is that developed under the technical part of the program described in Section 2.0. The goal was a boule which was 50 mm in diameter and 75 - 100 mm in total length parallel to [111]. This goal was achieved several times and permitted at least the suggested 30 laser rods to be obtained easily. It should be noticed that all of our boules were of sufficient length to obtain two sections; each section yielded laser rods depending on the internal quality. Under ideal circumstances, the highest yield would be 60 rods, i.e. 30 from each section if the boule were perfect.

In accordance with this contract, the rod fabrication process did not consist of unit operations which were developed years ago. Instead all rods were fabricated by the methods recently perfected under the initial part of this contract and described in a final report.[5] The reader is assumed to be familiar with the results of that effort. The basic fixture holds 16 rods and average rates of 12 rods/8 hour day were attained. A flow process for the rod fabrication steps is given in Figure 16. These steps were followed in the detail explained below. All rough rods were core drilled from the boule with a diamond drill to obtain starting material. Figure 17 shows several core drilled sections from contract boules.

3.2 Mounting of Rods

In preparation for the grinding and polishing operations, 16 rods are placed in the block in a horizontal position. This assembly is heated on a hot plate until the wax flows freely when placed on the rods. One end of the rods and then the other is removed from the block and coated with wax. The rods are rotated while being replaced in the block to spread the wax evenly. The block is placed on a flat glass with the rods in a vertical position and allowed to cool. The rods and feet are then lightly ground using a surface grinder. This operation generates a uniform surface during the final finishing operations and limits the amount of grinding required in subsequent steps. Figure 18 illustrates some polishing blocks with rods mounted in them.

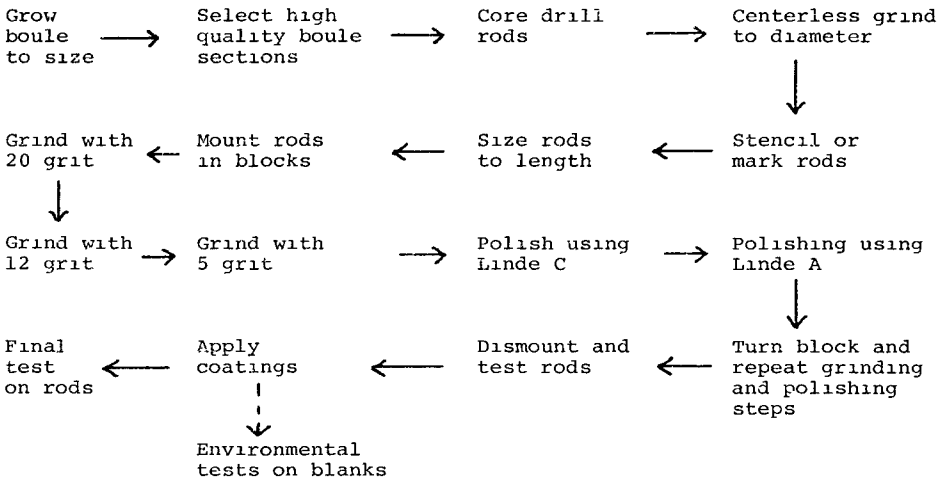


Figure 16 Flow Chart of Rod Manufacturing Process

Figure 17 Core Drilled Nd:YAG Sections

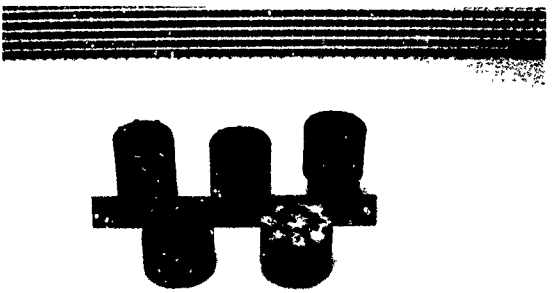


Figure 18 Polishing Blocks for Laser Rods

3.3 Twenty Micron Grinding

Select proper lap, mark grinding, and clean thoroughly using a mild detergent and water. Clean polishing machine to make sure all residual polishing compounds from previous operations are removed. Using a spherometer, measure the lap. If lap is not flat grind lap using 20 μm Al_2O_3 until flat. Install lap on polishing machine along with protective plastic ring. Using a comparator measure length of rods in block at several positions. Apply 50 grams of 20 μm Al_2O_3 to lap and add 50 ml of water. Mix until slurry is formed and distribute over lap. Using a timer, set proper lap rotation rate at 8 rpm. Place block of rods on lap and apply 80 strokes rotating block by 90° after each 20 strokes. (The term strokes applies to the operator moving the block across the lap and back. This is done in an approximate figure eight configuration.) Remove block from lap, measure length, and check surface finish. If grinding marks remain or rods are not working evenly replace block on lap. Continue grinding until a 20 μm surface finish is obtained and rods are being evenly worked (no point to point variation in rod length). Additional water can be added to the slurry to maintain the proper consistency. Approximately 0.002 inches is removed during this process. After completing run, clean block and lap thoroughly using a mild detergent and water.

3.4 Twelve Micron Grinding

Repeat steps of 12 μm grinding as required to obtain length. Apply 40 grams of 12μm Al_2O_3 to lap and add 40 ml of water. Mix until slurry is formed and distribute over lap. Use timer and set lap rotation rate of 8 rpm. Place block on lap and apply approximately 80 strokes rotating block by 90° after each 20 strokes. Remove block from lap, clean, measure lengths and check surface finishes. If proper surface finish has not been obtained or all block positions are not of equal lengths return block to lap and continue grinding. During grinding it may be necessary to apply more pressure to one side of the block than the other to get all rods of equal length. This can be accomplished by holding the block in place off center on the lap with the block side requiring additional material removal furthest from the center. Lap rotation may be increased to accelerate the process. When 12 micron finish is obtained and all rods are working evenly remove block and proceed to next step.

3.5 Five Micron Grinding

Repeat cleaning steps to assure that lap and block

are properly cleaned. Apply 25 grams of 5 μm Al_2O_3 to lap and add 40 ml of water. Mix to form slurry and distribute evenly over surface of lap. Use timer to set lap rotation rate to 8 rpm. Place block on lap and grind for approximately 400 strokes. This should take about 5 minutes. Again an equal amount of time should be spent in grinding in each of four positions 90° apart. The rod end faces are checked to make sure a 5 μm finish is obtained. The processing time is nominal since surface finish determines when process is complete. When proper surface finish is obtained clean block and tools using detergent and water and proceed to the polishing process.

3.6 One Micron Polishing

Select proper lap, marked polishing, and clean thoroughly using detergent and water. Clean work station to remove any residual polishing compounds from previous steps. Measure rod lengths using comparator. From this point on the polishing feet should be used for measurement to avoid any damage to rod end faces. Prepare slurry on a lap by mixing 10 grams of 1 micron Al_2O_3 to 50 ml of water. Approximately 5 ml of a suspension agent are added to reduce aglomerates. The slurry is mixed and spread evenly over the lap. Using timer set lap rotation rate of 8 rpm. Place block on lap and polish for 400 strokes rotating block by 90° after each 100 strokes. Remove block from lap, clean and inspect surface finish. If pits and scratches remain return to lap and repeat previous step by adding additional water to slurry as required. Continue polishing and inspecting until a surface finish free of pits is obtained and desired flatness achieved. Some scratches will remain at this point. The parts are now ready for the next step.

3.7 0.3 Micron Polishing

Repeat lap selection and cleaning. Prepare slurry by mixing one gram of 0.3 μm Al_2O_3 and 50 ml of water. Mix slurry and distribute evenly over lap. Using timer set lap rotation rate at 8 rpm rotating block by 90° after each 100 strokes. Place block on lap and polish for 400 strokes rotating block by 90° after each 100 strokes. Remove block from lap, clean and inspect surface finish. If scratching remains in excess of specification return to lap and continue polishing. Additional water can be added to the slurry as required to maintain proper consistency. Do not contact rod end faces during inspection as scratching may result. Continue polishing and inspecting until specified surface finish is obtained.

Rods are then cleaned and readied for second end grinding and polishing.

3.8 Second End Polishing

Install cover on block to prevent any damage to rod end faces already polished. Repeat grinding and polishing operations as outlined in sections 3.3 through 3.7. In last step of 3.7 check rod end face parallelism using Fizeau interferometer. When second ends are finished the block is thoroughly cleaned and rods are dismounted by heating the block on a hot plate until the wax softens and rods are easily slipped out. Rods are cleaned of residual wax, packaged and submitted for further testing.

3.9 Rod Coatings

Two coatings are specified for the laser rods delivered under this program. One rod end receives a single layer antireflection coating designed for 1.064 μm. This coating must have a reflection loss of less than 0.25%. The opposite end is dielectric coated to yield a reflectivity of 60 ± 3% at 1.064 μm. Note that the coatings on each end are distinguished by proper marking as indicated in Figure . The following procedures are used for a typical coating run.

Prepare a detergent solution and heat to boiling in pyrex dish. Take rods from boxes, inspect for obvious defects (chips, breakage, etc.). Put rods into the cleaning solution using the rod holder in the bottom of the dish. Let rods boil for approximately 5 - 10 minutes. While rods are boiling, check the vacuum station. Prepare coatings by melting. Clean the jar of loose coating. Check monitor for usable spot Clean base plate to remove loose coating or dirt. Clean container and fill with new full strength detergent. Prepare clean brush, wrenches, fixtures. After the rods have been boiling for the proper time, take one rod out at a time with a tweezer. Dip clean brush in detergent and brush end surface of rod while holding under water. Rinse in distilled water. Blow water off surface of rod using dry nitrogen. You must blow the water off in a single wave. Any other method of blow dry will leave water marks. Look at the rod surface with eye loupe and lamp. If the rod end is clean, place rod in rod tray under laminaire flow hood with ends to be coated facing in the same direction. When the rods are fully dry carefully place them in the proper size fixture. The end to be coated should be flush with the bottom of the fixture. After all rods are loaded carefully blow off all the ends with

dry nitrogen one more time to remove any dust or lint and place the protective top and bottom on the fixture. Keep the loaded fixture under the flow hood until it is loaded into the vacuum station. If the rod ends project outside of the fixture, it is essential to place a collar-guard-sleeve around the rods to protect them from breakage. Place small amount of foil over the collar to protect the ends that are not to be coated from receiving any extra coating. Take prepared fixtures to vacuum station, remove fixture bottoms, place in workholder. Insure that fixtures and rod ends do not overhang work holder and obstruct up and down motion of bell jar. See vacuum coating station procedures for pump down and coating information. While first batch of rods is in the pump down mode, prepare a second batch of rods. When first batch of rods is finished coating remove them and insert second batch. If rods are to be coated at both ends: Take rods out of the fixture and place uncoated end only into the solution. Repeat the cleaning procedure for these ends and load into fixtures. Water on coated end should not be harmful to a properly coated rod. Coat second end by following steps for pump down and coating. Remove from fixtures upon completion. Once again clean both ends and barrel with a very soft brush while being careful not to scratch the coating. Run a reflection curve on one rod and make copies. Make necessary entries into station log book.

During the coating process, batch samples are run for later evaluation of the coatings. These samples are usually of the same orientation, diameter, and subjected to the same fabrication procedures as the rod ends. The only difference is the length of the samples. Only a few mm are required and thus they are in the shape of a disk. These coated disks are utilized for subsequent environmental tests which may prove to be damaging to the laser rod. Among the tests are the following: Immersion, solubility, humidity, abrasion, and power handling. Since each of these tests is performed on a fresh sample, a group of six or more samples is prepared. Some photographs are given in the test reports of Section II. The disks are coated normally on only one side. They can be used several times by stripping the coating and repolishing.

Section III

Quality Control Tests

1.0 Introduction

The final major section of this report is intended to be a quality control manual which describes all tests utilized in the production of the contract item. The essence of this manual was composed as a test plan submitted to the U.S. Army prior to the completion of the confirmation and pilot production runs.

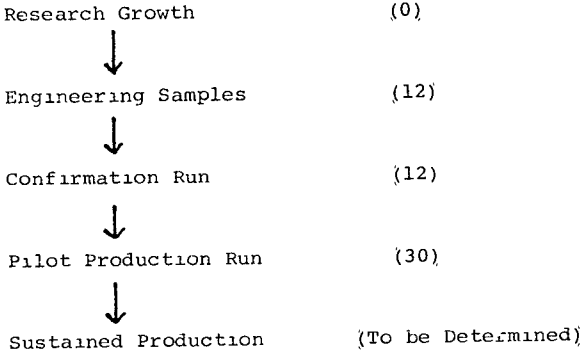
This test plan is prepared as a deliverable contract item (Item CLIN 0011, Exhibit H) of Contract No. DAAB07-77-C-0375, Modification P0003. The contract was issued in February, 1982 as a continuation program for the "Growth of Large Diameter Nd:YAG Laser Crystals". The objective of the contract is the study of growth techniques for achieving a boule "diameter" of two inches and a length of three inches at diameter. The boule should be capable of yielding 30 laser rods which meet the specifications of Appendice I - III. Thus this test plan describes tests and data which pertain to the fabricated laser rods primarily. The grown boule is subjected only to a minimum of tests which determine its suitability as raw material.

The test plan described herein is in preparation for the delivery of 12 laser rods to fulfill the Confirmation Run described in Contract Item No. SLIN 0009AB.

The plan will also utilize the same tests to fulfill Contract Item No. SLIN 0009AC which consists of the pilot run of 30 laser rods.

All growth, fabrication, and testing are performed at the plant facilities of Airtron, Division of Litton Industries, 200 East Hanover Avenue, Morris Plains, New Jersey. Supersvision of all program activities is conducted by Dr. Roger F. Belt. The U.S. Army Night Vision and Electro-optics Laboratory, Fort Belvoir, Virginia, is the contract sponsor. The designated program technical manager and recipient of all deliverable items is Ms. Kay Chloupek.

DESCRIPTIVE ITEMS



2.0 Time Schedule and Sequence of Tests

The approach and philosophy of our testing program will be based on the current manufacturing steps for boules and rods. These procedures were developed over a period of many years and are applied to all laser rods with allowance for deviations in specifications. The particular sequence we will adhere to is the AN/GVS-5 rod requirement. The current procedure is elucidated in Table IV which contains an abbreviated discussion of the manufacturing step, the description of each step, the estimated time for that manufacturing step and its in-process test, a list of all the respective test titles, and a reference to the test paragraphs. The latter refer to the test descriptions included as Appendices I - III.

In regard to our test program as formulated from the U.S. Army requirements and those of primary manufacturers which utilize the laser rod, we may note a few important differences. The manufacturer's specifications contain a test on fluorescent lifetime. Each rod must show a lifetime of 206-235 μs. This test is actually a confirmation of the neodymium level in the rod. It is repeatable and non-destructive. Therefore this test is performed on each rod in lieu of the X-ray emission or optical absorption of test samples located above and below the rods. One other test which is normally performed on some laser rods but is not specified is that of an extinction ratio. The normal value for this quantity is a minimum of 25 dB. The Army and manufacturer's specifications on the AN/GVS-5 rod do not require this test.

Table IV

Time Schedule and Sequence of Tests for

Nd:YAG Laser Rod Manufacturing

Step No.	Manufacturing Description	Time for Completion (Hrs) Manufac-Test	Test Title (1)	Test Reference (2)
1	Boule Growth	480 - 0.1	Boule Diameter	III, 4.2.1.1
			Boule Length	III, 4.2.1.2
2	Boule Inspection	0.1 - 0.1	Boule Inclusions	III, 4.2.2
3	End Cutting, Polishing	0.5 - 0	NTR	
4	Wafer Sampling	0.1 - 4	Nd Doping Level	I, 4.5.1 and 3.1
5	Boule Mapping	0.5 - 0.5	Boule Inclusions	III, 4.2.2
6	Core Drill Rods	1.0 - 0	NTR	
7	Centerless Grinding to Diameter	0.2 - 0.1	Rod Dimensions	I, 4.5.2 and 3.2
8	Cut and Grind to Length	0.4 - 0.2	Dimensions	I, 4.5.2 3.2
9	Stencil Rod Number, Mark AR End	0.1 - 0.1	Marking	I, 3.4
10	End Edge Bending	0.1 - 0.1	Chamfer	I, 3.2
11	Block in Fixtures	0.5 - 0	NTR	
12	First End Finish,	0.5 - 0.2	Surface Quality	I, 4.5.3.1 and 4.4.1.1
	Flatness Check		Surface Flatness	I, 4.5.3.2 and 3.3.1.2
13	Deblock, Cleaning Perpendicularity Check	0.5 - 0.2	NTR Perpendicularity	I, 4.5.3.4 3.3.1.4
14	Block for Second End	0.2 - 0	NTR	
15	Second End Finish, Parallelism Check	0.5 - 0.2	NTR Parallelism	I, 4.5.3.3 3.3.1.3 II, 1

Step No.	Manufacturing Description	Time for Completion (Hrs) Manufac Test	Test Title (1)	Test Reference (2)
16	Final Polishing	0.2 - 0.2	Surface Quality	I, 4.5.3.1 and 3.3.1.1
17	Deblock and Clean, Length Check	0.3 - 0.2 0.1 - 0.1	Dimension	I, 4.5.2 and 3.2
18	Quality Control Inspection			
	(a) Surface Finish	0 - 0.1	Surface Quality	I, 4.5.3.1
	(b) Extinction Ratio	0 - 0.2	Extinction Ratio	Undefined
	(c) Strain	0 - 0.1	Strain	I, 4.5.4.5 II, 2, 3
	(d) Dimensions	0 - 0.1	Dimensions	I, 4.5.2, 3.2
	(e) Lifetime	0 - 0.1	Lifetime	I, 3.1
	(f) Orientation	0 - 0	Orientation	I, 1.1
19	Rod Coating, Test Blanks	2 - 2.0 0.5 - 1.0		
	(a) Reflectivity	0.2 - 0.2	Reflectivity	I, 4.5.3.5
	(b) Power Handling	0.5 - 0.5	Power Handling	I, 4.5.3.6
	(c) Environmental	1.0 - 1.0	Environmental	I, 4.5.4
20	Inspection	0.2 - 0.2	NTR	
21	Package	0.1 - 0	NTR	
22	Shipping	0.2 - 0	NTR	

NOTES: (1) NTR = No test required

(2) See appendices

3.0 Description of Tests and Methods

In this section we give a brief description of each test performed on a boule or rod during the manufacturing cycle. The tests are listed in the order of Table I and a few words are said about the method which is used currently to perform the test.

Boule Diameter - This test measures the distance across a boule perpendicular to the [111] boule axis. A micrometer caliper is used.

Boule Length - This test measures the length at near constant diameter after the taper and bottom ends are removed. A micrometer caliper is used.

Boule Inclusions - This test determines harmful scattering sites due to iridium, gases, or other impurities. The method of test utilizes a highly collimated beam of light or a He-Ne laser for examination. The boule is also examined between crossed polarizers for micro and macro strain.

Neodymium Doping Level - This test determines quantitatively the amount of the lasing ion Nd^{3+} in the grown boule or rod. The test is conducted by extracting a disc of Nd:YAG above and below the rods. This disc is analyzed by comparison with known standards using X-ray emission, optical absorption, or fluorescent lifetime calibration curves.

Rod Dimensions - This test measures the length and diameter of the laser rod. Ordinary micrometers are employed.

Marking - This test identifies the rod serial number and the end of the rod with an AR coating. The test is visual.

Chamfer - This test checks the application of chamfered edges on the rod ends. The examination is by means of microscope.

Surface Quality - This test examines the end surface for scratches or digs after polishing. A microscope is used.

Surface Flatness - The test checks the end surface flatness. A standard optical flat is placed on the rod end.

Perpendicularity - This test establishes the perpendicularity deviation between the end face and the rod axis. An autocollimator is used for direct angular measurement after rod rotation of 360^{o} about its axis.

Parallelism - This test measures the parallelism of the two rod end faces. A Fizeau type interferometer is used with a He-Ne laser.

Orientation - This test identifies the rod axis as [111] Visual or X-ray methods are used.

Strain - This test measures the residual deviation from an optically perfect laser rod. The test is performed by means of a double pass Twyman-Green interferometer with a He-Ne light source.

Fluorescent Lifetime - This test measures the lifetime of the Nd^{3+} transition from $^4F_{3/2} - {}^4I_{11/2}$. It also confirms the amount of Nd^{3+} present in the rod. The test is performed on a rod by flashlamp excitation of the transition and detecting the emitted radiation decay by a detector and oscilloscope.

Reflectivity - This test measures the per cent reflectivity of the coated rod ends. The tests are performed on coated test blanks prepared with the laser rod end faces. Measurements are performed with a spectrophotometer.

Power Handling - This test determines the damage resistance of the end face coatings under irradiation with pulses from a Nd:YAG Q-switched laser. The tests are performed on coated blanks prepared with the rods.

Environmental Tests of Rod Coatings - These tests determine the durability of coatings applied to Nd:YAG test blanks. The blanks are coated simultaneously with the laser rods. Four different tests are performed on the coatings:

(a) Immersion test in methyl alcohol, ethyl alcohol, and acetone. These are for cleaning dust or dirt from optical surfaces.

(b) Solubility test is performed in a 4.5 weight % Na Cl solution for 24 hours.

(c) The humidity test is conducted at humidity conditions of 95 - 100% relative humidity at 120°F. The samples are preheated and tested at the conditions for 24 hours.

(d) Abrasion resistance is determined by rubbing a rubber pencil eraser across the optical surface.

Items (b), (c) and (d) are described fully by MIL-C-675.

4.0 Test Programs and Automation

The fabrication of high quality laser rods involves a series of steps which are not easily amenable to automated tests for production or quality control. Almost every operation in the cycle is performed manually on a single rod. The exceptions to this are only a few. They are the multiple block polishing of rods (Steps 11-12 of Table I) and the rod end face coatings (Step 19 of Table I). The block polishing can be performed on a group of 16 rods. However each rod of the group undergoes an individual test. Likewise the rod coating can be applied to a group of 16 rods in a separate fixture. No automated tests have been developed for the coatings on the rods. In fact the coating tests are performed on separate blanks of Nd:YAG which are prepared along with the rods.

No computer programs for any test procedures are in existence or used for production quantities desired at this time.

5.0 Reliability Testing

It should be stated that all tests described in our plan are classified as passive tests, i.e. they are not performed on the rods under actual lasing conditions. As a matter of experience in the use of Nd:YAG for military laser applications, no firm reliability models have been documented. It is tacitly assumed that when a laser rod meets all specifications described in this report, the rod will perform in an active laser. The tests are therefore necessary conditions but not sufficient to determine particular lasing performance.

5.1 Proposed Tests

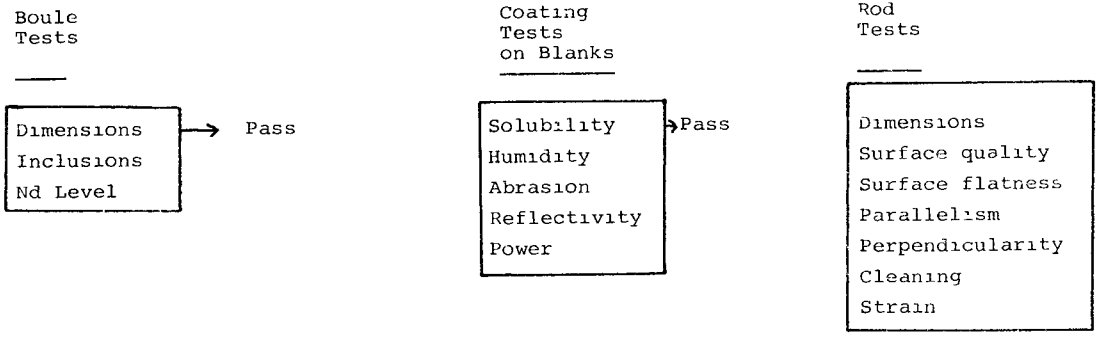
The proposed order of testing for reliability is the grown Nd:YAG boule, the end face coatings on the rod, and finally the rods themselves. It should be noticed that all testing is 100% on the boule and rods. The rod end face coatings are not 100% tested on each rod. The coating tests are performed on test blanks which are designed to represent the rods by statistics and identical preparation techniques. A flow diagram of the reliability Testing is given in Table II. All of these tests were described fully in Section 3.0 of this plan. The actual test procedures are presented in Section 6.0 of this plan.

5.2 Description of Tests for Inspection

Prior to the actual tests and inspection on the rods themselves, the following tests are to be performed. The grown boule is measured for dimensions, checked for inclusions, and the Nd doping level is determined by fluorescent lifetime X-ray, or optical absorption (Column 1 of Table II). Failure of any of these tests is a cause for rejection of the entire boule.

Table V

Reliability Testing Flow Chart



Test blanks are prepared next from wafer samples and shall be coated with the rods in the coating batch. The test blanks shall be tested in the following sequence: Solubility, Humidity, Abrasion resistance, Reflectivity, and Power handling. Failure of any test blank in any test is a failure of all rods in that coating batch. The tests on all rods shall consist of the tests specified in Column 2 of Table II and no failures shall be permitted. The final tests are to be performed on the rods themselves. These tests are given in the last column of Table II. In addition to these, the lifetime test may also be run on the rods to check for Nd level. Failure of any of these tests is a cause for failure of the rod. When batch polishing methods are used the procedures are different for the tests on perpendicularity and strain. These are given below for rods from boule sections.

(a) Perpendicularity - Two rods are randomly selected from the polishing batch. If both rods pass, all rods in the polishing batch are accepted. If both rods fail, all rods of the boule section will be rejected. If one rod fails, a second sample of two rods from the same boule section are selected. If both rods of second samples pass, all other rods of boule section are accepted. If one rod of second sample fails, all rods of boule section are rejected.

(b) Strain - Two rods randomly selected from each boule section shall be examined for strain. If both rods pass, all rods of the boule section will be accepted. If both rods fail, all rods of the boule section will be rejected. If one rod fails, a second sample of two rods from the same boule section are selected. If both rods of second sample pass, all other rods of boule section are accepted. If one rod of second sample fails, all rods of boule section are rejected.

It should be noticed that batch polishing is not a fixed requirement for this testing program. However it may be used in first end polishing to save time in production runs.

5.3 Requirements Based on Reliability Model

Since no definite model has been developed for the performance of a Nd:YAG rod, the requirements at the present time are arbitrary. Thus in the course of many years, some tests have been added and others deleted. These are dictated mainly by the use of a rod in a particular laser system. For all of these systems, there are no fixed or detailed reliability models. The basic assumption is that the Nd:YAG laser rod will perform if it meets all of the passive tests given in our test plan. From the viewpoint of experience and the production of more than 10^4 rods, the reliability of a rod is probably greater than 95%. This figure cannot be derived theoretically but is compiled from estimates of rejected rods or non performing systems.

5.4 Description of the Use of Test Data for Reliability

The use of our test data for reliability is based on the fact that 100% testing must be performed on the rod. If any test fails, that rod will be rejected. Thus the highest confidence level will be attained in active operation when all passive tests described in this plan are met. As explained before, the complete (100%) reliability of a rod generally cannot be attained or guaranteed solely from the passive tests.

If a rod fails a particular test, the rod may be recycled or fabricated again to meet that test. For example, any mistake in fabrication which does not destroy the laser rod, can be corrected and the rod re-tested to meet the chosen specification. The only test which completely fails a boule or rod and cannot be corrected is the Nd concentration. In such a circumstance the boule must be grown again with the proper amount of Nd.

It is particularly important to note that any of the rod coating tests which are performed on blanks, can be repeated; the repetition can be performed both on the blanks and rods by stripping the coatings, refabricating the pieces, and then recoating and testing until the specification is met. In this manner good Nd:YAG material is utilized fully and reliability is maintained at the highest level.

6.0 Test Procedures Used

This section contains a detailed description of all test procedures which will be used on the boule and laser rods. The sequence of tests follows the processes listed in Table I.

Boule Diameter - The seed end taper and boule end are removed from the grown boule by a diamond saw. The boule diameter is measured across a pair of opposing flats if the boule is not round. Measurements are made in a direction perpendicular to the [111] boule axis. The boule should measure 50 ± 5 mm. A measuring caliper with a least reading of at least 0.1 mm is used to make the measurement.

Boule Length - The length of the boule along [111] is measured after the end portions are cut and removed. The length shall measure 75 $^{+25}_{-0}$ mm exclusive of taper and end. A measuring caliper with a least reading of 0.1 mm is used to make the measurement.

Boule Inclusions and Strain - The end faces of the boule are polished to transmit a collimated beam of ordinary or laser light. The boule is first examined by projecting the beam parallel to [111] and observing any scattering in a perpendicular to [111] and looking down the axis. The boule is also examined between crossed polarizers for strain. Any areas of the boule which exhibit strain striae or inclusions shall be rejected for fabrication of laser rods. Inclusions of gases, solids, or other particulate matter larger than 1-5 um are not permissible in laser rods.

Boule Neodymium Doping Level - Concentrations of neodymium in the boule are determined by x-ray emission spectroscopy. Wafer samples are taken from the boule immediately above and below the region where rods are to be fabricated. Equipment is a Picker X-Ray Corp. spectrogoniometer with LiF crystal analyzer and appropiate scintillation counter having pulse height discrimination capabilities. Neodymium densities are measured using Nd L_α line. Rod doping densities are assigned based on the average of the values determined for the sample immediately above the rod and immediately below the rod. Appropriate Nd standards are used. High density powder pellets or polycrystalline standards are satisfactory. Secondary standards may be utilized. These include optical absorption or fluorescent lifetime which are run on boule pieces. Acceptable lifetimes for boules are in the range of 206-235 μs.

Neodymium Doping Level - The laser rod material shall consist of single crystal, neodymium doped yttrium aluminum garnet. Doping of the neodymium shall be 1.0 to 1.3 atomic percent substituted for yttria in the crystal. (Dopant density range: 1.38×10^{20} to 1.8×10^{20} ions per cm^3). Fluorescent lifetime to be between 206 and 235 μsec. Set up apparatus. Insert the test specimen in the apparatus, adjust oscilloscope vertical sensitivity such that peak of the fluorescent intensity represents a full scale deflection. Adjust horizontal scale to 100 μsec per division. With these conditions make an exposure of the resulting fluorescent intensity trace. Keeping all settings the same, replace the sample with a piece of clear YAG and make a second exposure on the same photograph to establish a baseline. Process the photographic record. From the photograph measure fluorescent intensity as a function of time. Calculate fluorescent lifetime from $t = (t_2 - t_1) \ln (I_1/I_2)$ where: t - Effective fluorescent lifetime of the sample; t_1, I_1 are the time and intensity respectively approximately 100 seconds after the onset of the pulse; t_2, I_1 are the time and intensity respectively at $t_2 = t_1 + 100$ μsec. Repeat the calculations of 3.1.2.8 for several time intervals. Acceptable parts will have fluorescent lifetimes that fall in the range of 206μ sec to 235μ sec.

Rod Dimensions - Diameter - 4.27 ± 0.02 mm. Length - 43.0 $^{+2.0}_{-0.0}$ mm. Clear Aperture - 4.00 ± 0.02 mm. Using a calibrated micrometer with a least count of 0.01 mm or less, measure the part diameter. Using a calibrated vernier caliper having a least count of 0.1 mm or less, measure the part length. Using the Nikon shadowgraph, measure the clear aperture of the part. Acceptable parts will be within the required tolerances on all dimensions measured.

Surface Quality - End surfaces shall be polished to a surface quality of 20-5. Using a binocular microscope and microscope illuminator, examine the surface under test for the presence of any scratches or digs. Where defects do exist, compare to scratch and dig standards for determination of size. Record number and sizes of defects observed. Acceptable parts will not exceed the number and sizes of defects allowed per MIL-O-13830.

Surface Flatness The ends shall be flat to within 0.2 wavelength of sodium light (5898Å). While rods are still in polishing block place the optical flat over the surfaces and observe the resulting fringe pattern. Perform under illumination at 5898Å. Acceptable parts will show fringe curvatures that are less than 0.4 fringes (0.2 waves).

Parallelism - The ends shall be optically parallel to within 10 arc seconds. Place rod under test in beam of interferometer. This can be done either while rods are still mounted in fixture or after dismounting. Final measurement is done on unmounted rods. Adjust the sample holder to produce the pattern with the minimum number of fringes. Acceptable parts will have less than 2.25 fringes across the clear aperture.

Perpendicularity - The ends shall be perpendicular to the rod axis within 5 minutes of arc. Place the rod under test in a vee block in the field of view of the autocollimator. Align the rod end surface with the autocollimator such that the reflection from the rod end face is seen in the autocollimator. Rotate the rod about its longitudinal axis and measure the total runout. Acceptable parts will have total runout of less than 10 minutes of arc.

Strain - No more than one half (1/2) strain free fringes per 43 mm of rod length are allowable when analyzed by double-pass Twyman Green interferometry. The rod under test is placed in the working arm of a Twyman Green interferometer. The working mirror is adjusted such that approximately three fringes are across the rod aperture. A photograph of the resulting fringe pattern is then taken. From this pattern the maximum curvature of the fringes is determined by measuring the average fringe spacing and the maximum deviation of the fringes from a straight line. The ratio of the deviation to the average spacing is calculated and expressed in units of fringes. Acceptable rods will have total distortions of less than 0.85 fringes.

Marking - Each rod shall have a serial number such that individual rods can be identified. The AR coated end of each rod shall also be identified with a dot or similar mark. Hold rod up to light and look through rod or examine diameter of rod under microscope illuminator. Look for serial number and dot under above conditions. Verify that dot is on antireflection coated end of rod. Acceptable rods will have a visible serial number and a dot at the AR end.

End Coating Reflectivity - One end surface shall be dielectric coated to have 60% ± 3% reflectivity for 1.0644 micron radiation. The opposite end shall be antireflection coated with low loss hard coating in accordance with MIL-C-675. This coating shall have a reflection loss of greater than 0.027% for 1.0644 micron radiation when in a medium with refractive index of 1.0. Wafer samples are coated with the lot of rods being processed. One sample is coated with the reflective coating lot and one with the antireflection coating lot. Using the Cary 14 Spectrophotometer with a reflectivity measuring attachment, a trace of the reflectivities of each of the samples is obtained. From these traces the reflectivity at 1.0644 is measured. Acceptable rods will be from reflective and antireflective coating lots that have measured reflectivities of 60% ± 3% and less than 0.25% respectively.

End Coating Cleaning - The coatings shall be capable of withstanding repeated cleaning and immersion in polar organic solvents without peeling, separating or changing in optical properties. Immerse coating witness sample in alcohol, remove and allow to dry. Repeat a minimum of six times. Then repeat and using methyl alcohol and then acetone. These immersions are to be performed for both reflective and antireflective coating samples. Acceptable coating lots will reveal no degradation in either visual appearance or optical characteristics.

End Coating Solubility Test - There shall be no visible evidence of film destruction after the coated rods are immersed for 24 hours in a sodium chloride solution. Prepare a salt water solution by mixing 6 ounces of common table salt (sodium chloride) per gallon of water. Immerse samples to be tested in solution for a period of 24 hours. Remove test samples from solution and wipe dry using lens tissue or a soft cloth. Acceptable parts will show no visible evidence of film destruction.

End Coating Humidity Test - There shall be no visible evidence of film deterioration after the coated rods are exposed for 24 hours to relative humidity of 95 to 100% at $120^{\circ} \pm 4^{\circ}F$. Establish humidity conditions of 95% to 100% at $120^{0} \pm 4^{\circ}F$ in temperature humdity chamber. Preheat samples to $120^{\circ}F$. Insert preheated samples in chamber and allow to remain for a period of 24 hours. Remove samples from chamber and wipe dry using lens tissue or soft cloth. Acceptable parts will show no evidence of film deterioration.

7.0 Identification of Instruments and Calibration

The measuring instruments which will be used for all of our testing are listed in Table III. These instruments were developed and put in service at Airtron over the last ten years of Nd:YAG development. Most of these are direct measurements and require no routine calibration. Some are used along with prepared standards. All tests are performed daily on the production line at Airtron. It should be noticed that tools and instruments from other sources, methods, or manufacturers can be used to perform the tests.

TABLE VI

Identification of Instruments and Calibration

NAME OF TEST	TEST ITEMS UTILIZED, SUPPLIES	PARTICULAR REQUIREMENTS	CALIBRATION REQUIREMENTS
Boule diameter	Mitotoyu Calipers Model 505-637	Least count of 0.1 mm or better	None
Boule length	Mitotoyu Calipers Model 505-637	Least count of 0.1 mm or better	None
Boule inclusions	He-Ne laser Model Bausch and Lamb Polarizers Model 31526260	None	None
Nd doping level	Picker X-ray, Model 3488K Cary Model 17 Spectrophotometer	Stability of ± 2%	Suitably prepared Nd standards
Rod dimensions	Starrett Model 483 V-Anvil Micrometer	Least count of 0.01 mm or better	None
	Nikon Model 6 Shadowgraph	Least count of 0.01 mm or better	
Chamfer	Nikon Model 6 Shadowgraph	Least count of 0.01 mm	None
Marking	Nikon Model 70702 binocular microscope and illuminator	10X magnification	None
Surface quality	Scratch & Dig Samples R.H. Beal Model 667 and viewing fixture Model 268	Per MIL-O-13830	None
Surface flatness	Optical flats	Flat to $\lambda/10$	None
Perpendicularity	Nikon Model 2100 Autocollimator	Resolution of 1 min of arc	None

TABLE VI (continued)

Identification of Instruments and Calibration

NAME OF TEST	TEST ITEMS UTILIZED, SUPPLIES	PARTICULAR REQUIREMENTS	CALIBRATION REQUIREMENTS
Parallelism	Fizeau Interferometer 632.8 nm laser source beam expansion optics viewing screen	Capable of positioning sample and measuring optical parallelism to 0.5 fringes (4 seconds)	None
Strain	Twyman-Green Interferometer Perkin Elmer Model 723	Capable of measuring wavefront distortion to $\lambda/10$	None
Lifetime	Xenon flashlamp and power supply	Flashlamp duration of	Standard Nd materials
	1.06 um blocking filter	Less than 0.0001% transmission at 1.06 u	
	Detector	S-1 surface	
	Oscilloscope - Tektronix Model 7704	Camera equipped Time base 100u sec/div.	
Orientation	Picker X-Ray or visual check	Measurement to $\pm 3^{O}$	None
Reflectivity	Cary Model 14 Spectrophotometer and reflectivity attachment	Capable of measuring reflectivities from 01.% to 100% at 1.06u	None
Cleaning	Beakers	10X Magnification	None
Solubility	Beakers	10X Magnification	None
Humidity	Tenny Model TH27 Temperature Humidity Chamber	Capable of attaining humidity at $120^{O}F \pm 4^{O}F$	None

TABLE VI (continued)

Identification of Instruments and Calibration

NAME OF TEST	TEST ITEMS UTILIZED, SUPPLIES	PARTICULAR REQUIREMENTS	CALIBRATION REQUIREMENTS
Abrasion	Rubber erasers	10X Magnification	None
Power handling	Q-switched Nd: YAG laser	100-500 MW/cm^2	Power density

8.0 Illustrations of Test Apparatus

This section concludes with a brief description of the normal test apparatus used for boule preparation, rod fabrication, and general quality control. The use of the apparatus is not given in detail since most of the items were described in previous reports. Small tools for dimensions are not shown because of their common availability. The discussion is arranged in order of use and follows the steps of Table IV.

Boule crystals and seeds are oriented along [111] by means of Laue X-ray back reflection. The analysis of Nd in boules is based on X-ray emission of primary standards. The equipment for both is a Picker system given in Figure 19. After laser rods are core drilled they are mounted in polishing blocks which hold up to 15 rods. Some of these are shown in Figure 20. Each block is processed at a single polishing station for the different grit sizes. A typical station is given in Figure 21. The end face flatness is determined by an optical flat. A group of these is pictured in Figure 22. The end face perpendicularity is measured with an optical autocollimator. A number of these are shown in Figure 23. The polished faces are checked microscopically with the assistance of a set of standard scratch and dig test examples given in Figure 24. All rods are fabricated with their ends parallel. The usual method is an in-process Fizeau interferometer located at each station. A similar instrument is given in Figure 25. For a rapid analysis of Nd, the fluorescent lifetime equipment of Figure 26 can be used directly on a finished rod. A final check of any rod optical distortion is performed by means of a Twyman-Green interferometer of Figure 27. The laser rod is coated by means of a vacuum evaporator with an optical thickness monitor. This is shown in Figure 28. A modern spectrophotometer is used to measure the reflectivity of the end coatings. The Cary instrument is pictured in Figure 29. For damage tests of the coatings, a Nd:YAG Q-switched laser is shown in Figure 30. All environmental tests of coatings are performed in a temperature-humidity chamber of Figure 31.

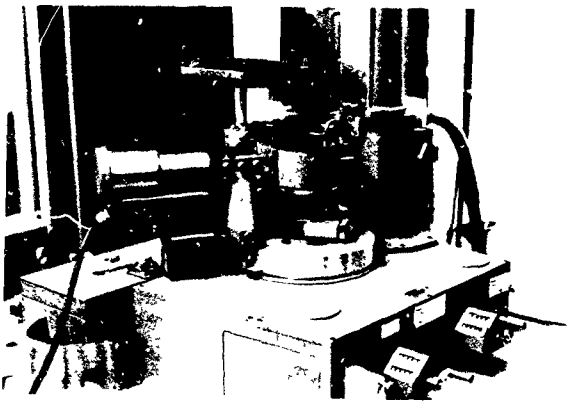


Figure 19 X-ray Diffraction and Emission

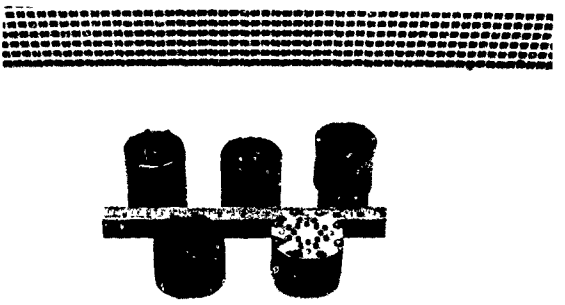


Figure 20 Blocks for Multiple Rod Fabrication

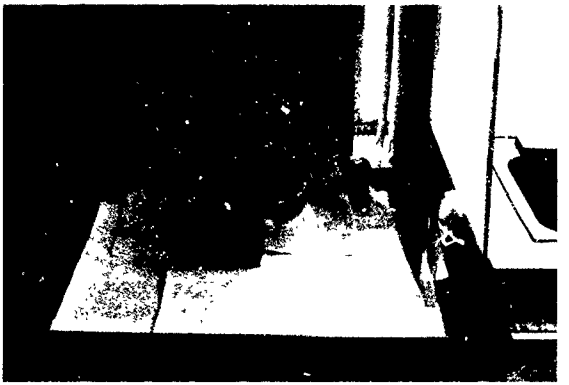


Figure 21 Single Polishing Station

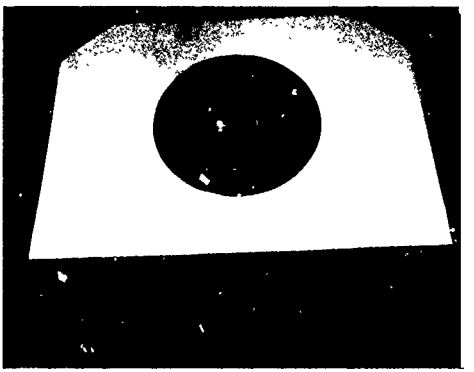


Figure 22 Optical Flats

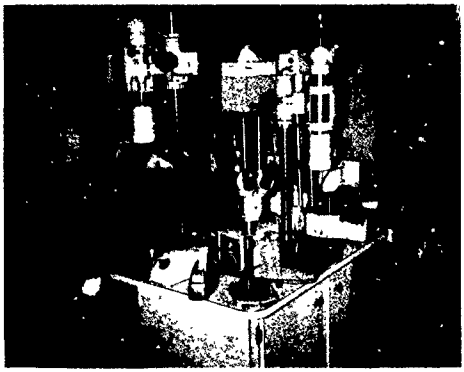


Figure 23 Group of Autocollimators

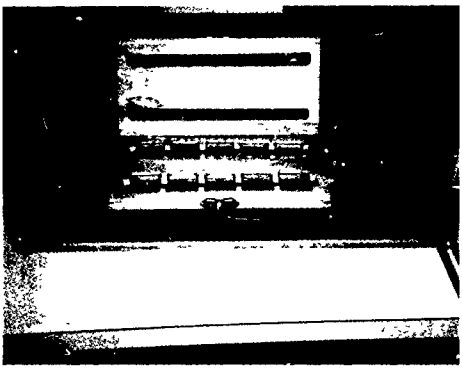


Figure 24 Scratch and Dig Standards

Figure 25 Interferometer

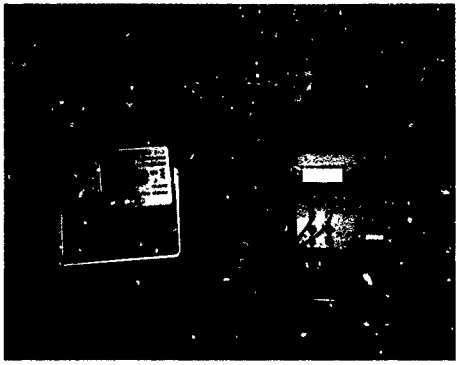


Figure 26 Fluorescent Lifetime Equipment

Figure 27 Twyman-Green Interferometer

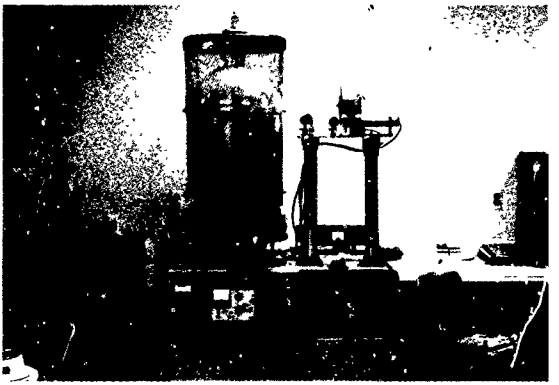


Figure 28 Vacuum Evaporation Station

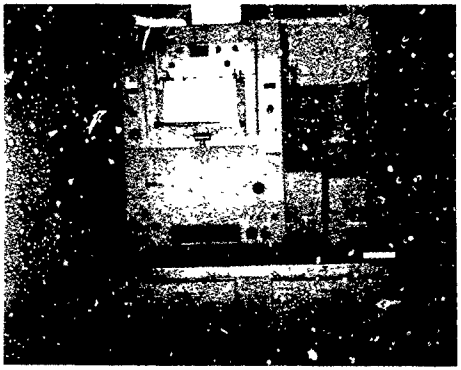


Figure 29 Cary Spectrophotometer

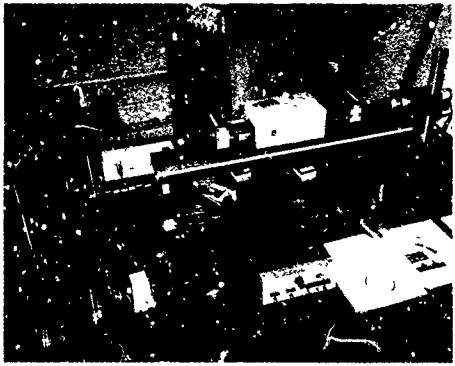


Figure 30 Nd:YAG Test Laser

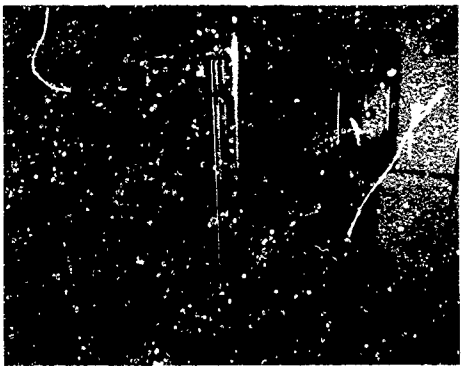


Figure 31 Environmental Chamber

APPENDIX I

ELECTRONICS COMMAND TECHNICAL REQUIREMENTS

SCS-507

NEODYMIUM DOPED YTTRIUM ALUMINUM GARNET LASER RODS

1. SCOPE

1.1 Scope.- This specification covers the requirements for single crystal <111> orientation, fine grind, neodymium doped yttrium aluminum garnet laser rods (Nd:YAG).

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on the date of the invitation for bids, form a part of this specification to the extent specified herein:

SPECIFICATIONS

MILITARY

MIL-C-675	Coating of Glass Optical Elements (Anti-Reflection).
MIL-O-13830	Optical Components for Fire Control Instruments; General Specifications Governing the Manufacture, Assembly and Inspection of.

2.2 In the event of conflict between this document and the referenced ones, the detail requirements of this specification shall take precedence.

3. REQUIREMENTS

3.1 Neodymium doping level.- The laser rod material shall consist of single crystal, neodymium doped yttrium aluminum garnet. Doping of the neodymium shall be 1.0 to 1.3 atomic percent substituted for yttria in the crystal. (Dopant density range: 1.38×10^{20} to 1.8×10^{20} ions per cm^3). (See 4.5.1).

3.2 Dimensions.- The laser rod shall have the finished dimensions and tolerances as specified in Figure 1. (See 4.5.2).

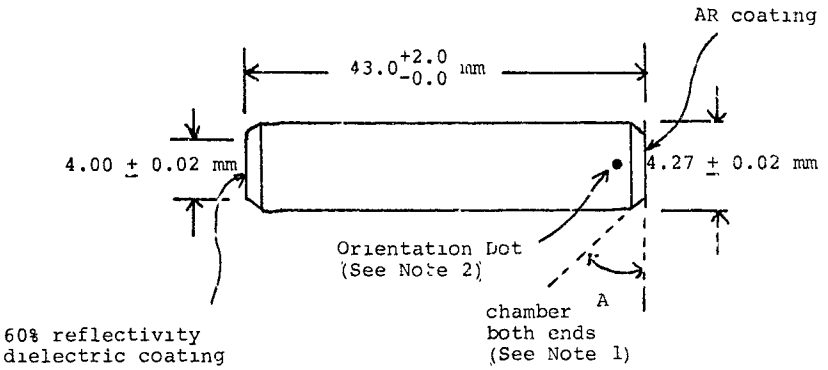


Figure 1

Notes: 1. Chamfer surface to be rough finish, 20 to 30 microinches. Chamfer angle A = 45° ± 5°.
2. The anti-reflection (AR) coated end of each rod shall be identified with a dot or similar mark located within 3 mm of rod end.

3.3 Optical properties.-

3.3.1 End surfaces.-

3.3.1.1 Surface quality.- End surfaces (figure 1) shall be polished to a surface quality of 20-5 (MIL-O-13830, Table I). (See 4.5.3.1).

3.3.1.2 Surface flatness.- The ends shall be flat to within 0.2 wavelength of sodium light (5898Å). (See 4.5.3.2).

3.3.1.3 Parallelism.- The ends shall be optically parallel to within 20 arc seconds. (See 4.5.3.3).

3.3.1.4 Perpendicularity.- The ends shall be perpendicular to the rod axis within 5 minutes of arc. (See 4.5.3.4).

3.3.2 End surface coatings.-

3.3.2.1 Cleaning of surface coatings.- The coatings shall be capable of withstanding repeated cleaning and immersion in polar organic solvents without peeling, separating or changing in optical properties. Refer to MIL-C-675. (See 4.5.4.1).

3.3.2.2 Solubility.- There shall be no visible evidence of film destruction after the coated rods are immersed for 24 hours in a sodium chloride solution. (See 4.5.4.2).

3.3.2.3 Humidity.- There shall be no visible evidence of film deterioration after the coated rods are exposed for 24 hours to relative humidity of 95 to 100% at 120° ± 4°F. (See 4.5.4.3).

3.3.2.4 Abrasion resistance.- There shall be no visible damage to the rubbed area of a coated surface after the ends of the coated rods are abraded. (See 4.5.4.4).

3.3.2.5 Power handling.- The coatings shall cover the entire clear aperture area of the surfaces and shall be capable of withstanding a minimum of 350 megawatts per square centimeter of laser power without degradation or change in optical characteristics. (See 4.5.3.6).

3.3.2.6 Reflectivity.-

3.3.2.6.1 As shown in figure 1, one end surface shall be dielectric coated to have 60% ± 3% reflectivity for 1.0644 micron radiation. (See 4.5.3.5).

3.3.2.6.2 The opposite end shall be anti-reflection coated with a low loss hard coating in accordance with MIL-C-675. This coating shall have a reflection loss no greater than 0.25% for 1.0644 micron radiation when in a medium with a refractive index of 1.0. (See 4.5.3.5).

3.3.3 Strain.- No more than one half (1/2) strain free fringes per 25.4 mm of rod length are allowable when analyzed by double-pass Twyman Green interferometry. (See 4.5.5).

3.4 Marking.- Each rod shall have a serial number such that individual rods can be identified. The AR coated end of each rod shall also be identified with a dot or similar mark in accordance with note 2, figure 1. (See 4.5.2).

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection.- Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements.

4.2 Classification of inspection.- Inspection shall be classified as follows:

(a) First article inspection.

(b) Quality conformance inspection.

4.3 Test plan.- The contractor prepared Government-approved test plan, as cited in the contract, shall contain:

(a) Time schedule and sequence of examinations and tests.

(b) A description of the method of test and procedures.

(c) Programs, if automatic test equipment is used, including flow charts and block diagrams.

(d) Identification and brief description of each inspection instrument with date of most recent calibration.

4.4 Inspection requirements -

4.4.1 First article inspection.- Prior to first article inspection, the following tests shall be performed: The Neodymium doping level (3.1 and 4.5.1) shall be performed on wafer samples from the boule. Failure of wafer samples is a failure of the entire boule. Test blanks prepared from wafer samples shall be coated with the rods in the coating batch. The test blanks shall be tested in the following sequence: Solubility (3.3.2.2 and 4.5.4.2), Humidity (3.3.2.3 and 4.5.4.3), Abrasion resistance (3.3.2.4 and 4.5.4.4), Reflectivity (3.3.2.6 and 4.5.3.5), and Power handling (3.3.2.5 and 4.5.3.6). Failure of any test blank in any test is a failure of all rods in that coating batch. First article tests on 10 rods shall consist of the tests specified in Table I and no failures shall be permitted.

Table I.- First article inspection

Inspection	Reqt Para	Test Para
Dimensions	3.2	4.5.2
Surface quality	3.3.1.1	4.5.3.1
Surface flatness	3.3.1.2	4.5.3.2
Parallelism	3.3.1.3	4.5.3.3
Perpendicularity	3.3.1.4	4.5.3.4
Cleaning of surface coatings	3.3.2.1	4.5.4.1
Strain	3.3.3	4.5.5

4.4.2 Quality conformance inspection.- Quality conformance inspection shall consist of tests specified for Group A inspection (Table II) and Group B inspection (Table III). The following shall apply:

(a) Prior to performing Group A inspection, the following inspections shall be done. The Neodymium doping level (3.1 and 4.5.1) shall be performed on wafer samples from each boule. Failure of wafer samples is a failure of the entire boule. Test blanks prepared from wafer samples shall be coated with the rods in the coating batch. The test blanks shall be tested in the following sequence: Solubility (3.3.2.2 and 4.5.4.2), Humidity (3.3.2.3 and 4.5.4.3), Abrasion resistance (3.3.2.4 and 4.5.4.4), Reflectivity (3.3.2.6 and 4.5.3.5), and Power handling (3.3.2.5 and 4.5.3.6). Failure of any test blank in any test is a failure of all rods in that coating batch.

Table II.- Group A inspection

Inspection	Reqt Para	Test Para	AQL
Dimensions	3.2	4.5.2	1%
Surface quality	3.3.1.1	4.5.3.1	
Surface flatness	3.3.1.2	4.5.3.2	
Parallelism	3.3.1.3	4.5.3.3	
Cleaning of surface coatings	3.3.2.1	4.5.4.1	

Table III.- Group B inspection

Inspection	Reqt Para	Test Para
Perpendicularity	3.3.1.4	4.5.3.4
Strain	3.3.3	4.5.5

4.4.2.1 Group B sampling.-

(a) Perpendicularity test - Two rods are randomly selected from each polishing batch. If both rods pass, all rods in the polishing batch are accepted. If both rods fail, all rods of the boule section will be rejected. If one rod fails, a second sample of two rods from the same boule section are selected. If both rods of second sample pass, all other rods of boule section are accepted. If one rod of second sample fails, all rods of boule section are rejected.

(b) Strain test - Two rods randomly selected from each boule section shall be examined for strain. If both rods pass, all rods of the boule section will be accepted. If both rods fail, all rods of the boule section will be rejected. If one rod fails, a second sample of two rods from the same boule section are selected. If both rods of second sample pass, all other rods of boule section are accepted. If one rod of second sample fails, all rods of boule section are rejected.

4.5 Test Methods.-

4.5.1 Neodymium doping level.- Concentrations of neodymium in the laser rods shall be determined using x-ray emission spectroscopy. Wafer samples taken from regions of the boule immediately above and below the region where rods are to be fabricated, see figure 2, shall be subjected to analysis and comparison with standards. Equipment utilized shall include Picker X-Ray Corp., GE-XRD-5 or equivalent spectrogoniometers with LiF crystal analyzers and appropriate scintillation counters having pulse height discrimination capabilities. Neodymium densities shall be measured using Nd L_{α} line. Rod doping densities will be assigned based on the average of the values determined for the sample immediately above the rod and immediately below the rod, figure 2. If not already available to the contractor, he shall prepare appropriate standards for accomplishing measurements. If prepared by the contractor, the standards shall be formed in accordance with established laboratory procedures which serve to insure homogeneity of the standards and densities approaching that of a single crystal. High density powder pellets or polycrystalline standards may be considered with the above boundaries. In determining the Nd concentrations of the calibration samples, comparison to all methods will be made: wet chemistry analysis, x-ray analysis and/or optical density measurements. The standard shall be maintained during the course of the contract and secondary standards utilized if desired or necessary.

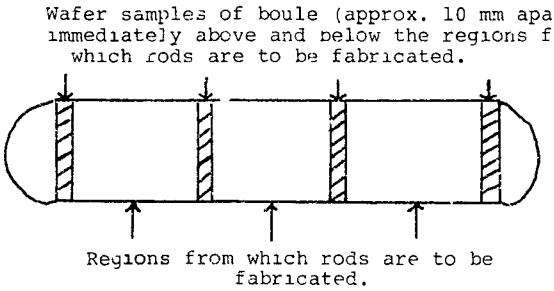


Figure 2

4.5.2 Dimensions.- Rod dimensions shall be measured using suitable mechanical, electrical or optical devices with appropriate care being exercised as to not damage the face surfaces. The rod shall be inspected for presence of required serial number and orientation dot.

4.5.3 End surfaces.-

4.5.3.1 Surface quality.- Surface quality of each rod end shall be determined in accordance with MIL-O-13830.

4.5.3.2 Surface flatness.- Surface flatness of each rod end shall be established using a master optical flat of at least 1.0 inch in diameter and certified flat over that diameter to 0.1 wavelength for 5898Å radiation.

4.5.3.3 Parallelism.- End face parallelism of each rod shall be established using a Fizeau interferometric device with a HeNe (6328Å) source. Conversion from fringe count to arc seconds will be accomplished. Final parallelism will be determined on unstressed rods.

4.5.3.4 Perpendicularity.- End face - rod axis perpendicularity of each rod will be established using a suitably calibrated auto-collimator in conjunction with a mechanical mounting apparatus for rotating the rods through 360°. Since parallelism has been established, one end face is aligned with the auto-collimator and zeroed. The rod run-out is established by rotating the rod through 360° about its axis and using the reticle of the collimator to determine the magnitude. Final perpendicularity will be determined on unstressed rods.

4.5.3.5 Reflectivity.- During rod end-face coating, suitable test blanks prepared from wafer samples shall be simultaneously coated for use in determining the reflectivity values of the rod coatings. Measurements will be performed on a Cary 14 Spectrometer or equivalent.

4.5.3.6 Power handling.- Prior to the power handling test, the end surface coatings shall meet all reflectivity specifications. The test blanks prepared from wafer samples will be utilized to ascertain the power handling capabilities of the coatings for 1.06 micron radiation. The anti-reflection coating shall meet MIL-C-675. The coatings shall be tested to determine if they can withstand a minimum of 350 megawatts per square centimeter of laser power without degradation or change in optical characteristics. The appropriate coating under test will be inserted into the beam path of a "Q" switched laser operating at a wavelength of 1.06 microns,

at a position such that a power density of 350 megawatts per square centimeter is incident on the coating. The area under test shall be equivalent to the rod polished aperture and be fully illuminated with the "Q" switched pulse. The laser pulse width shall be 15 ± 5 nanoseconds full width at half maximum which places an average energy per pulse of 0.660 joules on the test laser. The samples after irradiation shall meet the specifications for reflectivity cited in 3.3.2.6 and exhibit no burn spots or crazing or signs of the surface coating lifting from the substrate.

4.5.4 End surface coatings.-

4.5.4.1 Cleaning of surface coatings.- Each rod shall be immersed in ethyl alcohol, methyl alcohol and acetone. Once the rod is removed and allowed to dry it shall be re-immersed. Each rod shall receive a minimum of 6 immersions and dehydrations in each of the three solvents. Upon analysis, there should be no evidence of peeling, separating or changing in optical properties.

4.5.4.2 Solubility.- Each blank shall be subjected to the solubility test specified in 4.6.8 of MIL-C-675.

4.5.4.3 Humidity.- Each blank shall be subjected to the humidity test specified in 4.6.9 of MIL-C-675.

4.5.4.4 Abrasion resistance.- Each blank shall be subjected to the abrasion resistance test specified in 4.6.11 of MIL-C-675.

4.5.4.5 Strain.- Each rod will be examined in a double pass Twyman Green interferometer using a HeNe (6328Å) laser source. The rods shall be unstressed in mount. The fringe per inch value shall be determined neglecting edge fringing effects.

5. PREPARATION FOR DELIVERY

5.1 The contractor shall package the rods in a suitable container to preserve the end face conditions and the general integrity of the rods during shipment and storage.

6. NOTES

6.1 A fine grind outside surface roughness is defined as being in a range of 20 to 30 microinches finish.

APPENDIX II

US ARMY ELECTRONICS RESEARCH & DEVELOPMENT COMMAND
NIGHT VISION & ELECTRO-OPTICS LABORATORY
FT. BELVOIR, VA

AMENDMENTS TO SCS 507 DATED 12 NOV 1975

"NEODYMIUM DOPED YTTRIUM ALUMINUM GARNET LASER RODS"

CHANGES: Jun 1979

1. Paragraph 3.3.1.3 entitled "PARALLELISM" shall be upgraded to read:

"The ends shall be optically parallel to within 10 arc seconds. (See 4.5.3.3)"

2. Paragraph 3.3.3 entitled "STRAIN" shall be upgraded to read:

"No more than one-half (1/2) strain free fringe per 43 mm of rod length are allowable when analyzed by double pass Twyman Green interferometry."

3. Paragraph 4.5.5 entitled "STRAIN" shall be changed to:

"Each rod will be examined in a double pass Twyman Green interferometer using a HeNe (6328Å) laser source. The rods shall be unstressed in the mount during final test. The fringe per mm value shall be determined neglecting edge fringing effects."

4. All previous references to paragraphs 3.3.1.3, 3.3.3 and 4.5.5 in SCS 507 dated 12 Nov 75 shall now reflect the amended values.

APPENDIX III

US ARMY ELECTRONICS RESEARCH & DEVELOPMENT COMMAND

NIGHT VISION & ELECTRO-OPTICS LABORATORY
FT. BELVOIR, VA

ND:YAG LARGE DIAMETER BOULE SPECIFICATION
JUNE 1979

1. SCOPE

1.1 Scope. This specification, taken with SCS-507 dated 12 Nov 1975 and amended Jun 1979, covers the detail requirements for single crystal [111] orientation, fine grind, neodymium doped yttrium aluminum garnet laser rods (Nd:YAG).

2. APPLICABLE DOCUMENTS

2.1 Laser Rod Specification. SCS-507 dated 12 Nov 1975 and amended Jun 1979 form a part of this specification. The subject documents are included therein.

3. REQUIREMENTS

3.1 Single Crystal Boule Description. Single crystal Yttrium Aluminum Garnet doped with Neodymium shall be grown from a melt contained in iridium crucibles using the Czochralski method. These single crystal boules shall be the source of rough cut solid-state Nd:YAG laser rods.

3.1.1 Boule Orientation. Boules are grown along [111] crystal axis. They may be cylindrically shaped or possess (211) and (110) side facets. A central core is present and misoriented from the remainder of the boule.

3.1.2 Boule Composition - Nd Doping Level. Boules shall contain a neodymium doping level of 1.38×10^{20} to 1.8×10^{20} ions per cm^3 over their usable length which shall begin after "seed" to diameter taper is completed.

3.1.3 Boule Dimensions.

3.1.3.1 Length - Boules shall have at least 75 mm, +25 mm, -0 mm of uniform diameter material parallel to [111] exclusive of taper or end.

3.1.3.2 Diameter - Boules shall measure 50 mm $\pm$ 5 mm perpendicular to [111].

3.1.3.3 Core - A single core in the boule center having a maximum diameter of 4 mm is allowable.

3.2 Boule Optical Quality.

3.2.1 Striae. When examined between crossed polarizers normal to the [111] growth axis, striae may be observed. When examined along the growth axis no striae will be observed. Since no quantitative value for maximum allowable striae is known, the striae shall be characterized descriptively if that portion of the boule is used for laser rods.

3.2.2 Strain. Boule strain, viewed between crossed polarizers, delineates the boule core and faceting regions. No rods shall be fabricated from these areas. Laser rod strain requirements of SCS 507 amended June 79 shall prevail.

3.2.3 Inclusions. Small defects due to gases, iridium, or other crystal phases, capable of scattering light in transmission through the boule shall be identified and no laser rods fabricated from regions of the boule containing inclusions.

4.0 QUALITY ASSURANCE PROVISION

In as much as the end item of this production process development is a completely fabricated Nd:YAG laser meeting all the specifications and requirements of SCS-507, amended Jun 1979, only paragraphs 3.1.3 and 3.2.3 of the Nd:YAG Boule Specification dated Jun 1979 need be inspected for with respect to the crystal boule. All previous Quality Assurance Provisions included in SCS-507, amended Jun 1979 are applicable.

4.1 Confirmatory Sample Inspection. Confirmatory sample tests on one (1) "Large Diameter Nd:YAG Single Crystal Laser Boule" from which a minimum of twelve (12) laser rods complying with SCS 507, amended Jun 1979, are fabricated shall be performed. Requirements 3.1.3.1 and 3.1.3.2 performed in accordance with 4.2.1 and 3.2.3 performed in accordance with 4.2.2 shall be met by the boule.

4.1.2 Pilot Run Inspection. The Pilot Run shall meet the following inspection requirements: One (1) section of a "Large Diameter Nd:YAG Single Crystal Laser Boule" shall yield a minimum of thirty (30) laser rods complying with SCS 507 amended Jun 1979. Requirements 3.1.3.1 and 3.1.3.2 performed in accordance with 4.2.1 and 3.2.3 performed in accordance with 4.2.2 shall be met by the boule.

4.2 Test Methods.

4.2.1 Dimensions - Boule dimensions shall be measured using suitable mechanical or optical devices with appropriate care as to not damage the boule.

4.2.1.1 Diameter. The minimum boule diameter dimension shall be measured perpendicular to [111] after the taper and end of the as-grown crystal are removed. Both ends of the boule will be measured and they shall meet the requirements. If the boule shape is faceted, then the diameter dimension shall be taken from the flattened region.

4.2.1.2 Length. The minimum boule length shall be met after all cutting and polishing of any inspection surfaces perpendicular to [111].

4.2.2 Inclusions - The as-grown boule will be examined with a high intensity microscope illuminator perpendicular and parallel to the crystal growth direction. Areas of the boule exhibiting scattered illumination will be identified and avoided in fabrication. Laser rods will contain no observable inclusions.

Appendix IV

Engineering Sample Test Report

The first delivery of this contract was prepared during the second interim report period. The delivery consisted of twelve engineering sample laser rods which were to be obtained from a single Nd:YAG boule. These rods were not required to meet the proposed specifications in their entirety. However for reference purposes we reproduce in Table I an abbreviated set of the contract laser rod specifications. These were derived from U.S. Army SCS 507 Documents plus selected amendments to the present program (See Appendices I, II, III) Essentially these rods conform to the AN/GVS-5 type rod specification.

The boule from which the laser rods were extracted was N2533. A picture is given in Figure 1. This boule was one of the first to give good growth results. It contained only a few blossoms in the taper region and several near the end of growth. Otherwise the quality was reasonably good. More than 12 rods were core drilled from the rough boule. However the best rods (as judged by optical and visual testing) were selected for complete fabrication. The fabrication was performed in the Airtron laser rod shop by regular personnel using techniques and procedures in daily use. Table II is a summary of the test data which were accumulated during and after the fabrication. These data meet the proposed specifications of Table I in every category. A few of the rods contained slight scattering sites of submicrometer size. These sites did not affect the total strain as exhibited by the Twyman-Green fringe pattern. Thus there should be little or no effect on the active operation. It appears that all of our passive tests indicate that rod quality is fully equal to that obtained from smaller diameter boules.

Figure 1 View of Boule N-2533 between Crossed Polarizers. The middle and bottom sections yielded strain free rods.

Table I

Laser Rod Specifications from
SCS 507 and Amendments

Characteristics	Specification	Test Method
Nd Dopant	1.0 - 1.3 atomic %	Fluorescent Lifetime ($220 \pm 15\mu s$)
Dimension	Length 43.0 mm $^{+2}_{-0}$mm	Calipers
	Dia. 4.27 ± 0.02mm	Micrometer
End Surface Quality	20 - 5	Comparison Standards
End Surface Flatness	$\lambda/5$	Optical Flat
Parallelism	10 sec	Fizeau Interferometer
Perpendicularity	5 min	Autocollimator
Strain	<0.5 Fringe/43mm	Twyman Green - Double Pass
End Coating	End 1: $60 \pm 3\%$ R	Cary Spectrophotometer
	End 2: AR with <0.25% loss	

Table II

Summary of Test Data for Twelve Engineering Sample Laser Rods from Boule N-2533

Rod Number	Diam. (mm)	Length (mm)	Perpen. (min)	Parallelism (sec)	Lifetime (μs)	Scatt. Sites	Strain (Total fringe)
R4071	4.27	45.8	2.5	<10	206	2	<0.1
R4072	4.27	45.8	2.0	<10	210	10	<0.1
R4073	4.26	45.8	2.5	<10	201	10	0.1
R4074	4.27	45.7	2.5	<10	218	20	0.2
R4075	4.26	45.8	2.5	<10	210	10	0.2
R4076	4.27	45.8	0.5	<10	208	5	<0.1
R4077	4.28	45.9	2.0	<10	215	5	<0.1
R4079	4.27	45.8	2.0	<10	219	<5	<0.1
R4080	4.27	44.3	1.5	<10	212	2	<0.1
R4081	4.28	45.8	1.5	<10	206	1	0.4
R4082	4.27	45.6	1.0	<10	214	<5	0.2
R4083	4.27	45.9	2.0	<10	204	<5	0.3

Appendix V

Confirmatory Sample Test Report

1.0 Introduction

These test results are transmitted as a deliverable contract item to fulfill the Confirmation Run described in Contract Item No. SLIN 0009AB, of Contract No. DAAB07-77-C-0375, Mod. P0003. The contract was issued in February, 1982 as a continuation program for the "Growth of Large Diameter Nd:YAG Laser Crystals." The objective of the contract is the study of growth techniques for achieving a boule "diameter" of two inches and a length of three inches at diameter. The boule should be capable of yielding 30 laser rods which meet the pertinent specifications. Thus this report describes tests and which pertain to the fabricated laser rods primarily. The grown boule is subjected only to a minimum of tests which determine its suitability as raw material.

The tests in this report were described fully in a preliminary Test Plan which was forwarded to the technical contract monitor on Sept. 1, 1982. This Test Plan will not be repeated here but forms a necessary adjunct to the test results.

All growth, fabrication, and testing were performed at the plant facilities of Airtron, Division of Litton Industries, 200 E. Hanover Ave., Morris Plains, NJ. Supervision of all program activities was conducted by Dr. Roger F. Belt. The U.S. Army, Night Vision and Electro-Optics Laboratory, Fort Belvoir, Virginia is the contract sponsor. The designated program technical manager and recipient of all deliverable items is Ms. Kay Chloupek/Mr. Jeff Paul.

2.0 Testing Sequence and Results

The boule growth of Nd:YAG is the first process step and only a few short tests are performed at this stage. These tests qualify the boule for further processing into laser rods. The majority of the testing occurs at the rod manufacturing stage and is interspersed with these operations. Finally the coatings are applied to the rods and any test blanks to be evaluated. The following paragraphs describe the full range of testing on each of the specified items.

2.1 Boule Description and Tests

The particular boule chosen for our Confirmatory Samples was 2572. This boule was grown under the contract with 1.1% atomic % Nd. It was grown at a pull rate of .013 inches per hour and a rotation rate of 15 rpm. A 4.5 x 4.5 inch cylindrical iridium crucible was utilized. A picture of this boule taken in ordinary light is given in Figure 1. A routine examination of the boule between crossed polarizers showed that the internal quality was high and free of strain except for a small blossom near the end of the growth. The top tapered seed end and the bottom end with the blossom were removed with a diamond saw. A picture of the boule at this stage is given in Figure 2. The ends were polished and a detailed optical examination was performed for scattering, strain, and other defects. The boule was free of these and two tiers of laser rods could be obtained from the boule. The boule was marked for core drilling and 21 rod lengths were drilled

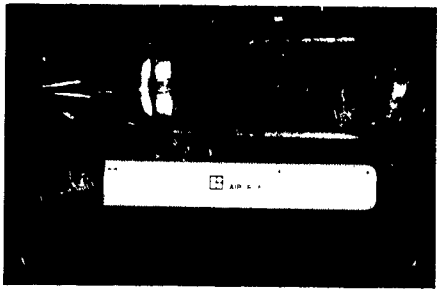


Figure 1 Boule from Run N2572.
Ordinary Light.

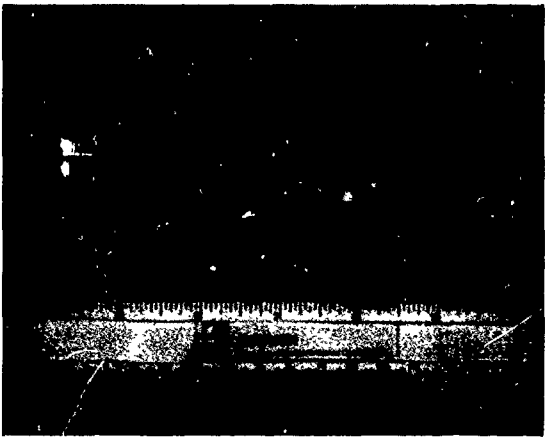


Figure 2 Boule from Run N2572, Top and Bottom Ends Removed, Ordinary Light.

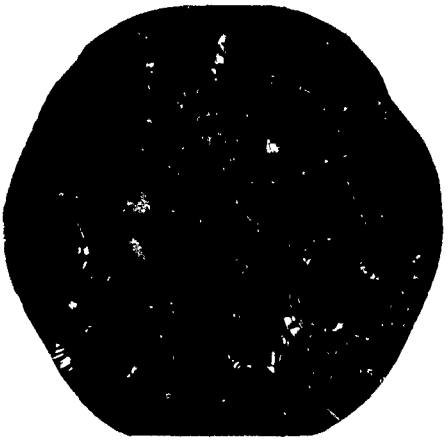


Figure 3 Bottom View of Boule After Core Drilling of 21 Lengths for Rods.

from the boule. The bottom of the boule is pictured in Figure 3 after the core drilling operation. The 21 rods lengths were sufficient for 42 test rods after the lengths were divided in half. After this procedure was done, the individual rods were examined optically. It was found that some of these were imperfect. Actually 37 of the rods were satisfactory and sent through the remaining process steps.

A summary of all the boule tests is given in Table I. The Nd concentration of the boule was determined by a method similar to that of the rods. At the top and bottom of the boule, small lengths were core drilled and the fluorescent lifetimes were measured by decay curves. These lifetimes were converted to a weight % Nd (and hence atomic %) with the aid of a calibration curve given in Figure 4. The latter was prepared from standards whose Nd concentration was determined originally by x-ray and optical absorption. Data were supplied by Airtron and the U.S. Army Electronics Command at Fort Monmouth, N.J.

2.2 Coating Tests Performed

In accordance with our test plan, all coating tests are related to specific environmental conditions. The tests, except for immersion are not performed on rods but on Nd:YAG test blanks which are prepared along with a group of rods. The type of coatings under discussion and preparation are a single layer (quarter wave optical thickness) of MgF_2 for the 0.25% R end, and a multilayer oxide-sulfide combination for the 60% R end.

Table I

Results of Boule Tests

Name of Test Performed	Test Reference (Table I of Test Plan)	Required Specification	Measured Results
Boule Diameter	III, 4.2.1.1	50±5 mm	Bottom, 50.7 mm Top, 48.0 mm
Boule Length	III, 4.2.1.2	75 + 25 mm - 0	95.3 mm
Nd Concentration	I, 4.5.1 and 3.1	1.0-1.3 atomic %	Bottom 1.30% Top 0.96%
Boule Quality/ Inclusions	III, 4.2.2	No strain or inclusions	Free of strain and inclusions
Orientation	I, 1.1	[111]	[111]

Test blanks were prepared from scrap Nd:YAG boules. They were formed in the shape of a cylindrical disc of 4-7mm diameter x 2-3mm high. The samples were cut with the major faces perpendicular to [111]. Thus the test samples were of the same orientation as the original laser rods. The blanks were polished mechanically using the very same procedures as the laser rod end faces. Finally the blanks and rods experienced the same conditions of coating variables and furthermore had an identical layer on each sample.

A summary of the environmental testing is given in Table II. Some comments on each of the tests are now in order.

2.2.1 Six coated Nd:YAG pieces were used for these tests (Fig. 5). The samples were Nd:YAG 0.25-0.50 inches in diameter by 0.2-0.5 inches thick. One face was polished and coated using the same processes used for the laser rods. The second surface was rough ground. For spectral testing absorbing material could be applied to the ground surface and O/D to eliminate interferences.

2.2.2 The materials required for performance of these tests included tape, cheesecloth, acetone, ethyl alcohol and trichlorethylene and a 5% Na Cl solution (6 oz. Na Cl per gallon of water.) The abrasion tester consisted of the Eraser Abrasion Coating Tester of D7680606 with the eraser end completely covered by a 1/4" thick by 3/8" wide pad of clean, dry, laundered cheesecloth conforming to CCC-C-440. The cheesecloth is secured to the tester with an elastic band. The environmental chamber was capable of 95% to 100% relative humidity at 120°F. Environmental chamber was capable of a temperature range of -80°F to + 160°F. A Cary 14 spectrophotometer and reflectance attachment was used for coating reflectivity.

2.2.3 The following tests on abrasion and humidity were performed using two 15watt fluorescent tubes as the light source and the unaided eye. The surface to eye distance shall not exceed 18 inches.

1. Examine the coating for coverage of the entire clear aperture.

2. Examine the coating for evidence of flaking, peeling, cracking, blistering, stains, smears, discolorations, spatter, holes, scratches, digs or other defects.

3. Press a 1/2" wide strip of tape firmly against the coated surface.

4. Quickly remove at an angle normal to the surface.

5. Inspect for any coating removal.

6. Place the samples in the humidity chamber at 120°F and 95%-100% relative humidity.

7. Remove samples from chamber after 24 hours and clean coated surfaces by lightly wiping surfaces with a soft tissue wetted with ethyl alcohol.

8. Inspect for any evidence of coating degradation - flaking, peeling, stain smears, etc.

9. Within 1 hour of cleaning, rub the surface of each sample with the cheesecloth covered abrader a minimum of 50 strokes at 1 lb. pressure. Hold the sample with tweezers during these tests.

10. Inspect each surface for evidence of coating degradation.

11. Wrap each sample in lens tissue and store for further testing.

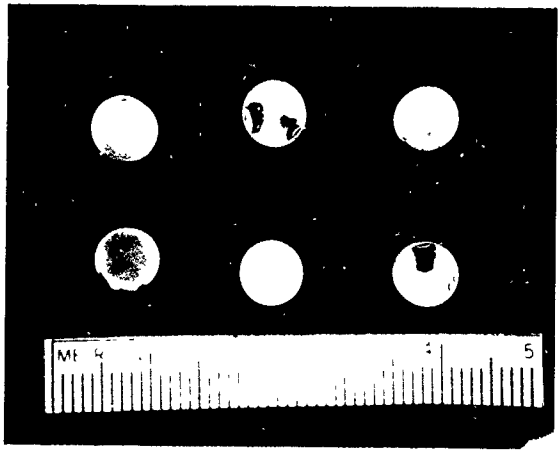


Figure 5. View of Six Coated Samples of Nd:YAG Used for Environmental Tests (Chips and defects are on opposite end of coating.)

2.2.4 The following tests were performed on temperature cycling and immersion.

1. Place the 6 samples in the temperature test chamber and reduce the temperature to -80°F at a rate not to exceed 4°F/minute.

2. Hold the temperature at -80°F ± 2°F for 2 hours.

3. Increase the temperature to + 160°F at a rate not to exceed 4°F/minute.

4. Hold at 160°F for 2 hours.

5. Return to room temperature at a rate of 4°F/minute.

6. Remove the samples from the test chamber.

7. Inspect coating for evidence of degradation - peeling, flaking.

8. Prepare individual beakers containing trichloroethylene, acetone, and ethyl alcohol at room temperature. The beakers are to contain sufficient amounts of solvent to cover the samples when immersed.

9. Immerse samples in trichloroethylene for at least ten minutes.

10. Remove sample and allow to evaporate to dryness.

11. Repeat for acetone.

12. Repeat for ethyl alcohol.

13. Clean each coated surface using clean cheesecloth moistened with ethyl alcohol.

14. Examine the coating for any evidence of degradation.

2.2.5 The solubility test in a salt spray solution was performed as follows:

1. a 5% by weight salt solution was prepared by dissolving 6 ounces of NaCl in a gallon of water.

2. Samples were immersed in the salt solution for 24 hours at 20°C.

3. The samples were removed, rinsed in pure water and inspected.

2.2.6 The spectral tests are given in Fig. 6 for an actual laser rod.

1. The coatings are applied to ground surface and O.D. samples to eliminate unwanted reflections.

2. Place sample coated surface down in Cary 14 spectrophotometer with reflectance attachments and measure reflection at specified wavelength.

3. Remove sample from spectrophotometer and insert spacer ring of same nominal thickness as the piece under test.

4. Replace sample with ground and blackened side down and measure reflectance at the specified wavelength.

5. Calculate coating reflectivity.

2.3 Rod Tests

All of the rod tests prescribed are performed during the manufacturing steps of the rod. Some are completed after the rod has been fabricated and coated. Table III lists all of the rod tests in the order of performance. A brief summary of the measured results is also listed.

A complete set of all individual rod tests is listed in Table IV for the 12 delivered confirmatory samples. It can be seen that these measurements fall within the required specifications.

Table IV

Test Results For Confirmatory Sample Laser Rod Delivery

Rod Number	Rod Diameter (mm)	Rod Length (mm)	Surface Quality	Surface Flatness (λ)	Parallelism (sec)	Perpendicularity (min)	Strain (Fringes)	Strain (Fringes/mm) $X10^{-3}$	Lifetime (μs)	Wt % Nd	Nd Conc (Atom %)
4130	4.267	44.907	20-5	< 1	<10	2.5	0 2	4.4	229	.81	1.11
4131	4.267	44 882	20-5	<.1	<10	3	0.5	11.1	223	87	1.20
4132	4 267	44 247	20-5	< 1	<10	5	0.2	4.4	236	.73	1 00
4133	4 267	44 933	20-5	< 1	<10	2.5	0.5	11.1	227	.83	1.14
4134	4 267	44.882	20-5	< 1	<10	2	0 3	6.7	235	74	1.02
4135	4 267	44.933	20-5	< 1	<10	2	0 1	2.2	217	.93	1.29
4136	4.267	44 882	20-5	<.1	<10	2	0.5	11 1	229	.81	1.11
4137	4.267	44.882	20-5	<.1	<10	2 5	0 4	8.9	229	.81	1.11
4139	4 267	44 882	20-5	<.1	<10	3.5	0.5	11 1	232	.78	1.07
4141	4.267	44.933	20-5	<.1	<10	3.5	0 5	11 1	229	.81	1 11
4142	4 267	44 882	20-5	<.1	<10	4 5	0 4	8 9	219	91	1 25
4143	4 267	44.882	20-5	< 1	<10	4	0.5	11 1	236	73	1 00

3.0 Conclusions

It has been demonstrated that large diameter Nd:YAG boules can be grown with a high yield of good quality laser rods. The boule of the confirmatory sample lot gave 42 laser rods of (4.3 x 43.0mm) dimensions. Thirty-seven rods gave passable optical quality while thirty-three met all specifications. Twelve rods were chosen for delivery. These rods were subjected to extensive coating and fabrication tests. All rods meet the contract specifications and design goals.

4.0 Delivery of Rods

Twelve fabricated and tested laser rods were delivered to the U.S. Army, Night Vision and Electro-Optics Laboratory, Fort Belvoir, Virginia. The delivery was made by personal carrier to Dr. A. Pinto/J. Paul on November 23, 1982.

Appendix VI

Pilot Production Test Report

1.0 Introduction

These test results are transmitted as a deliverable contract item to fulfill the Pilot Production Run described in Contract Item No. SLIN 0009AC, of Contract No. DAAB07-77-C-0375, Mod. P0004. This contract was issued in February, 1982 as a continuation program for the "Growth of Large Diameter Nd:YAG Laser Crystals". The objective of the contract is the study of growth techniques for achieving a boule "diameter" of two inches and a length of three inches at diameter. The boule should be capable of yielding at least 30 laser rods which meet the required specifications. Thus this report describes tests and data which pertain particularly to the fabricated laser rods. The grown boule is subjected to a minimum of tests which determine its suitability as raw material.

The tests in this report were described fully in an approved Test Plan which was forwarded to the technical contract monitor on September 1, 1982. This Test Plan will not be repeated here but forms a necessary adjunct to the test results.

All growth, fabrication, and testing were performed at the plant facilities of Airtron, Division of Litton Industries, 200 East Hanover Ave., Morris Plains, New Jersey. Supervision of all program activities was conducted by Dr. Roger F. Belt. The U.S. Army, Night Vision and Electro-Optics Laboratory, Fort Belvoir, Virginia is the contract sponsor. The designated program technical manager and recipient of all deliverable items is Dr. A. Pinto.

2.0 Testing Sequence and Results

The boule growth of Nd:YAG is the first process step and only a few short tests are performed on the boule. These tests qualify the boule for further processing into laser rods. The majority of the testing occurs at the rod manufacturing stage and is interspersed with these operations. Finally the coatings are applied to the rods and test blanks are evaluated. The following paragraphs describe the full range of testing on each of the specified items.

2.1 Boule Description and Tests

The particular boule grown for the Pilot Production Run was N3122. This boule was formulated with 1.1% atomic % Nd. An experimental pull rate of 0.020 inches per hour and a rotation rate of 15 rpm were employed. A 4.5 x 4.5 inch cylindrical iridium crucible was utilized. A picture of this boule taken in ordinary light is given in Figure 1. A routine examination of the boule between crossed polarizers showed that the internal quality was high and free of strain. No blossoms were present along the entire length. The top tapered seed end and the bottom end were removed with a diamond saw. A normal core was found. A picture of the boule at this stage is given in Figure 2 taken between crossed polarizers. The ends were polished and a detailed optical examination was performed for scattering, strain and other defects. The boule was free of these and two lengths of laser rods could be obtained from the boule. The

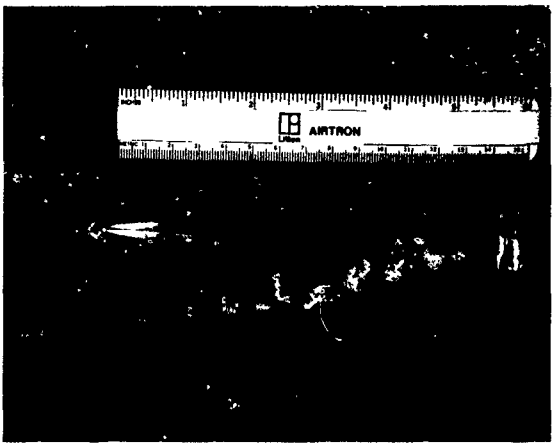


Figure 1. Single crystal boule of Nd:YAG for pilot production run. Boule N3122, photograph taken in ordinary light.

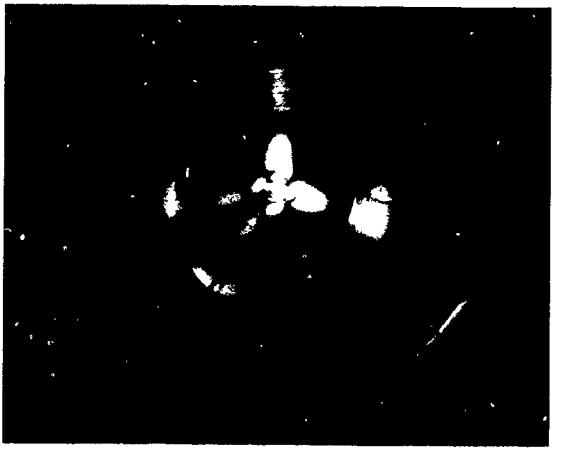


Figure 2. Boule N3122 photographed between crossed polarizers after removing top and bottom and polishing ends. The normal central core along [111] and slight strain are present. Boule length is 95mm.

boule was marked for core drilling and 38 rods were drilled from the bottom of the boule. In addition 26 rods were core drilled from the top of the boule, Figure 3. After this procedure was done, all individual rods were examined optically. It was found that some of these were imperfect. Actually most of the rods were satisfactory and sent through the remaining process steps of fabrication.

A summary of all the boule tests is given in Table I. The Nd concentration of the boule was determined by a method similar to that of the rods. At the top and bottom of the boule, small lengths were core drilled and the fluorescent lifetimes were measured by decay curves. These lifetimes were converted to a weight % Nd (and hence atomic %) with the aid of a calibration curve given in Figure 4. The latter was prepared from standards whose Nd concentration was determined originally by x-ray and optical absorption. Data were supplied by Airtron and the U.S. Army Electronics Command at Fort Monmouth, N.J.

2.2 Coating Tests Performed

In accordance with our test plan, all coating tests are related to specific environmental conditions. The tests, except for immersion are not performed on rods but on Nd:YAG test blanks which are prepared along with a group of rods. The type of coatings under discussion and preparation are a single layer quarter wave optical thickness of MgF_2

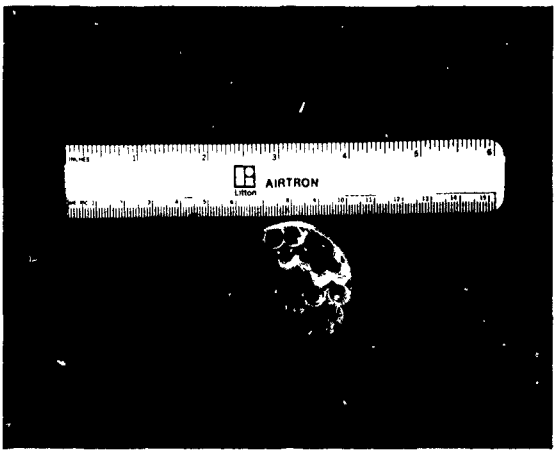


Figure 3. Top section of Boule N3122. This photograph was taken during the core drilling operation. Seventeen rods were core drilled already and nine more were marked on the section.

Table I

Results of Boule Tests on N3122

Name of Test Performed	Test Reference (Table I of Test Plan)	Required Specification	Measured Results
Boule Diameter	III, 4.2.1.1	50 ± 5mm	Bottom, 48.8mm Top, 46.5mm
Boule Length	III, 4.2.1.2	75 + 25mm	95.0mm
Nd Concentration	I, 4.5.1 and 3.1	1.0 - 1.3 atomic%	Bottom 1.33% Top 0.96%
Boule/Quality Inclusions	III, 4.2.2	No strain or inclusions	Free of strain and inclusions
Orientation	I, 1.1	[111]	[111]

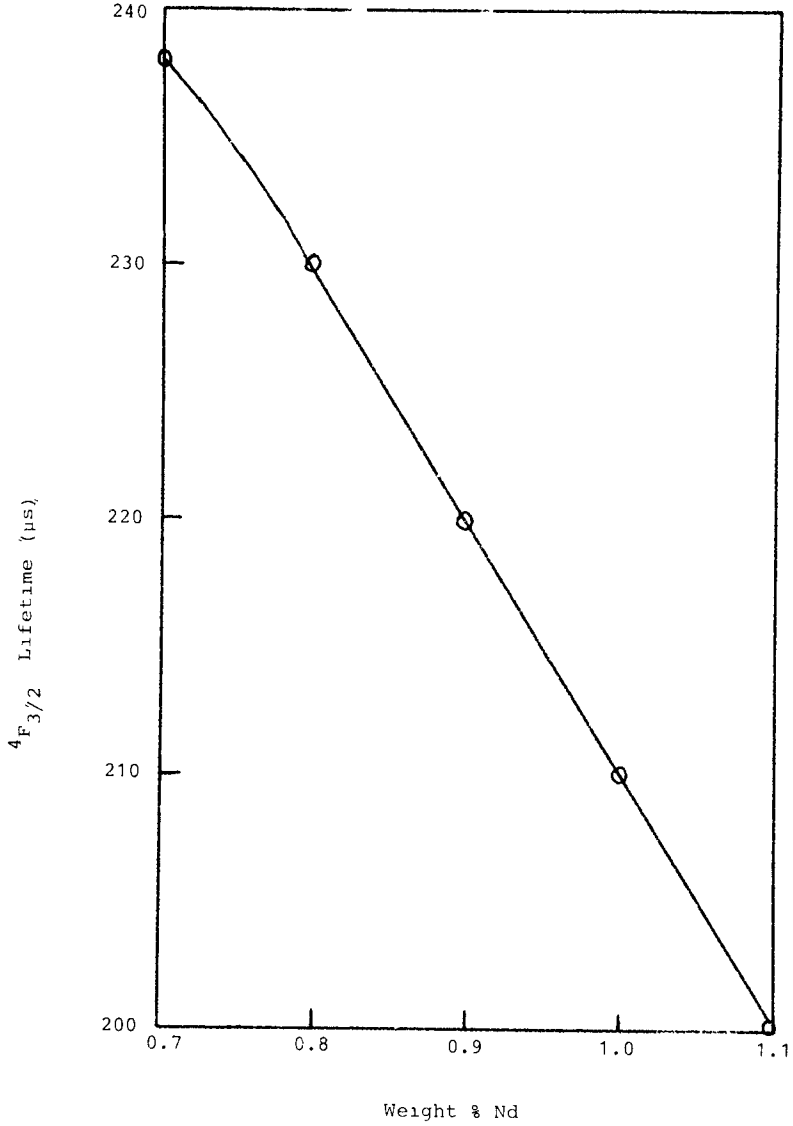


Figure 4. Fluorescent Lifetime as a Function of Nd Concentration, Calibration Curve from Data of U.S. Army Electronics Command

for the 0.25% reflection end, and a multilayer oxide-sulfide combination for the 60% reflection end.

Test blanks were prepared from scrap Nd:YAG boules. They were formed in the shape of a cylindrical disc of 4-7mm diameter x 2-3mm high. The samples were cut with the major faces perpendicular to [111]. Thus the test samples were of the same orientation as the original laser rods. The blanks were polished mechanically using the very same procedures as the laser rod end faces. Finally the blanks and rods experienced the same conditions of coating variables and furthermore had an identical layer on each sample.

A summary of the environmental testing is given in Table II. Some comments on each of the tests are now in order.

2.2.1 Six coated Nd:YAG pieces were used for these tests (Figure 5). The samples were Nd:YAG 0.25-0.50 inches in diameter by 0.2-0.4 inches thick. One face was polished and coated using the same processes used for the laser rods. The second surface was rough ground. For spectral testing absorbing material could be applied to the ground surface and O.D. to eliminate interferences.

2.2.2 The materials required for performance of these tests included tape, cheesecloth, acetone, ethyl alcohol and trichorethylene and a 5% NaCl solution (6 oz NaCl per gallon of water). The abrasion tester consisted of the Eraser

TABLE II

RESULTS OF ENVIRONMENTAL TESTS ON COATED BLANKS

Name of Test	Test Reference (Table I of Test Plan)	Required Specification	Measured Results
Immersion	I, 4,5,4,1 3,3,2,1	No peeling, separation or change of optical properties	No change of optical properties
Solubility	I, 4,5,4,2 3,3,2,2	No film destruction after 24 hours	No film change
Humidity	I, 4,5,4,3 3,3,2,3	No film change after 24 hours	No peeling or cracking
Abrasion	I, 4,5,4,4 3,3,2,4	No damage to coating after test	No damage
Reflectivity	I, 4,5,3,5 3,3,2,6	60 $\pm$ 3% R for end 1; <0.25% R for end 2	60 $\pm$ 3% R < 0.25% R
Power Handling	I, 4,5,3,6 3,3,2,5	350 MW/cm^2 with no damage	No damage at 350 MW/cm^2

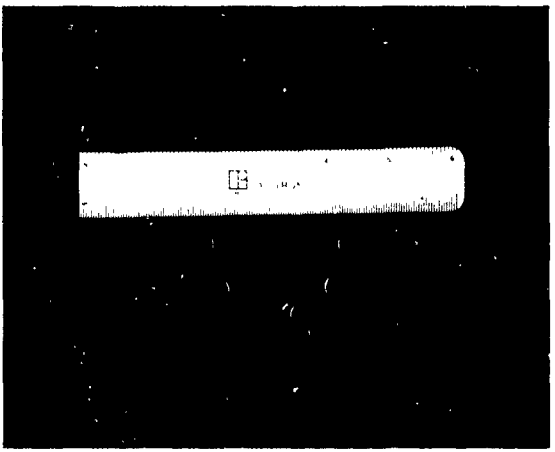


Figure 5. View of samples of Nd:YAG used for environmental tests. Samples are oriented and coated in vacuum chamber along with laser rods.

Abrasion Coating Tester of D7680606 with the eraser end completely covered by a 1/4" thick by 3/8" wide pad of clean, dry, laundered cheesecloth conforming to CCC-C-440. The cheesecloth is secured to the tester with an elastic band. The environmental chamber was capable of a temperature range of -80°F to +160°F. A Cary 17 spectrophotometer and reflectance attachment was used for coating reflectivity.

2.2.3 The following tests on abrasion and humidity were performed using two 15 watt fluorescent tubes as the light source and the unaided eye. The surface to eye distance shall not exceed 18 inches.

1. Examine the coating for coverage of the entire clear aperture.
2. Examine the coating for evidence of flaking, peeling, cracking, blistering stains, smears, discolorations, spatter, holes, scratches, digs or other defects.
3. Press a 1/2" wide strip of tape firmly against the coated surface.
4. Quickly remove at an angle normal to the surface.
5. Inspect for any coating removal.
6. Place the samples in the humidity chamber at 120°F and 95%-100% relative humidity.
7. Remove samples from chamber after 24 hours and clean coated surfaces by lightly wiping surfaces with a soft tissue wetted with ethyl alcohol.

8. Inspect for any evidence of coating degradation - flaking, peeling, stain smears, etc.

9. Within 1 hour of cleaning, rub the surface of each sample with the cheesecloth covered abrader a minimum of 50 strokes at 1 lb. pressure. Hold sample with tweezers during these tests.

10. Inspect each surface for evidence of coating degradation.

11. Wrap each sample in lens tissue and store for further testing.

2.2.4 The following tests were performed on temperature cycling and immersion.

1. Place the 6 samples in the temperature test chamber and reduce the temperature to -80°F at a rate not to exceed 4°F/minute.

2. Hold the temperature at -80°F +2°F for 2 hours.

3. Increase the temperature to +160°F at a rate not to exceed 4°F/minute.

4. Hold at 160°F for 2 hours.

5. Return to room temperature at a rate of 4°F/minute.

6. Remove the samples from the test chamber.

7. Inspect coating for evidence of degradation - peeling, flaking.

8. Prepare individual beakers containing trichloroethylene, acetone, and ethyl alcohol at room temperature. The beakers are to contain sufficient amounts of solvent to cover the samples when immersed.

9. Immerse samples in tricholorethylene for at least 10 minutes.

10. Remove sample and allow to evaporate to dryness.

11. Repeat for acetone.

12. Repeat for ethyl alcohol.

13. Clean each coated surface using clean cheesecloth moistened with ethyl alcohol.

14. Examine the coating for any evidence of degradation.

2.2.5 The solubility test in a salt spray solution was performed as follows:

1. A 5% by weight salt solution was prepared by dissolving 6 ounces of NaCl in a gallon of water.

2. Samples were immersed in the salt solution for 24 hours at 20°C.

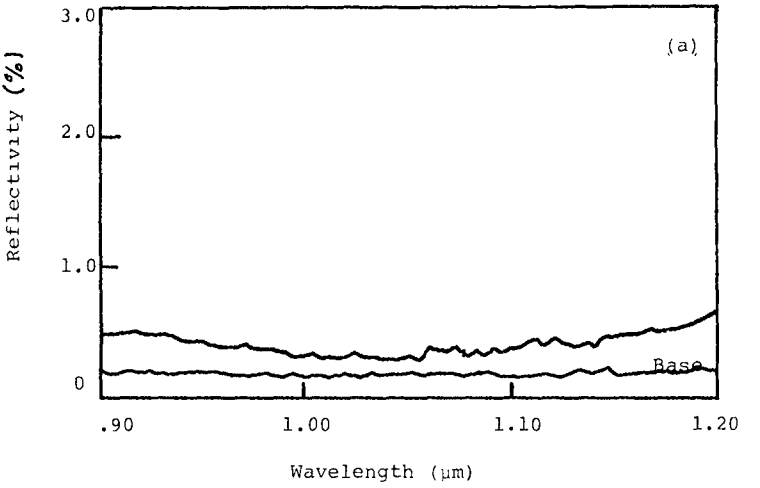
3. The samples were removed, rinsed in pure water and inspected.

2.2.6 The spectral tests are given in Figure 6 for an actual laser rod.

1. The coatings are applied to ground surface and O.D. samples to eliminate unwanted reflections.

2. Place sample coated surface down in Cary 17 Spectrophotometer with reflectance attachments and measure reflection at specified wavelength.

3. Remove sample from spectrophotometer and insert spacer ring of same nominal thickness as the piece under test.



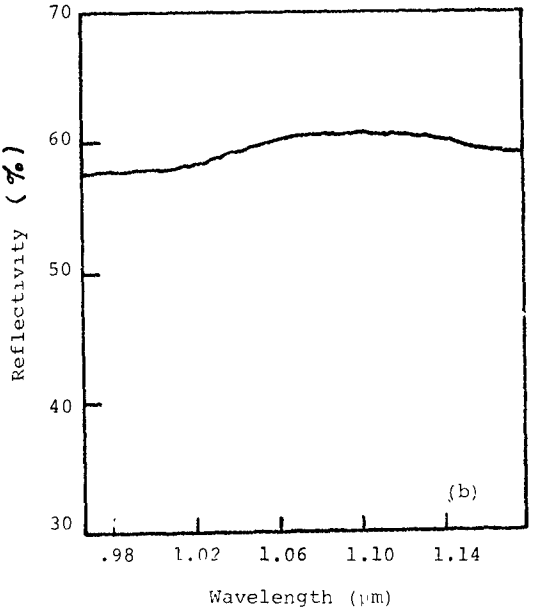


Figure 6 Reflectivity as a Function of Wavelength for Actual Laser Rod. (a) Anti-reflection Coated End of Rod (b) Nominal 60% Reflectivity End of Rod

4. Replace sample with ground and blackened side down and measure reflectance at the specified wavelength.

5. Calculate coating reflectivity.

2.3 Rod Tests

All of the rod tests prescribed are performed during the manufacturing steps of the rod. Some are completed after the rod has been fabricated and coated. Table III lists all of the rod tests in the order of performance. A brief summary of the measured results is also listed.

A complete set of all individual rod tests is listed in Table IV for the 30 delivered Pilot Production samples. It can be seen that these measurements fall within the required specifications.

3.0 Conclusions

It has been demonstrated that large diameter Nd:YAG boules can be grown with a high yield of good quality laser rods. The boule of the Pilot Production sample lot gave over 60 laser rods of (4.3 x 43.0mm) dimensions. All fabricated rods gave passable optical quality and met all specifications. Thirty rods were chosen for delivery. These rods were subjected to further fabrication tests. All rods meet the contract specifications and design goals.

4.0 Delivery of Rods

Thirty fabricated and tested laser rods were delivered to the U.S. Army, Night Vision and Electro-Optics Laboratory, Ft.

Table III

Results of General Tests on Rods

Name of Test	Test Reference (Table I of Test Plan)	Required Specification	Measured Results (See Table IV for Details)
Rod Dimensions	I, 4.5.2 and 3.2	Length $43.^{+2}_{-0}$ mm	43^{+2}_{-0} mm
		Diam. $4.27^{\pm}$.2 mm	$4.27^{\pm}$.2 mm
Surface Quality	I, 4.5.3.1	Quality of 2-5	20-5
	I, 4.4.1.1	No scratches/digs	No digs
Surface Flatness	I, 4.5.3.2	<0.2 λ at 590 nm	<0.1
	I, 3.3.1.2		
Parallelism	I, 4.5.3.3	<10 arc sec at 632 nm	<10 sec
Perpendicularity	I, 4.5.3.4	<5 min	<5 min
Strain	I, 4.4.4.5	<0.5 fringe/rod	<0.5 fringe
	II, 2.3	Double pass	
Lifetime	I, 3.1	205-238μs	214-238μs
Orientation	I, 1.1	[111] $\pm$ 2°	[111] $\pm$ 2°

Table IV
Test Results for Pilot Production Run Laser Rod Delivery

Rod Number	Rod Diameter (mm)	Rod Length (mm)	Surface Flatness (λ)	Parallelism (arc sec)	Perpendicularity (min)	Strain (Fringes)	Strain (Fringes/mm) $X10^{-3}$	Lifetime (μs)	Wt % Nd	Nd Conc. (Atom %)
R7084	4.27	44.856	≤.1	10	2 5	≤.2	≤ 4.4	226	83	1 14
R7085	4.27	44.602	≤ 1	10	3 5	≤.2	4.5	215	.95	1 31
R7086	4.27	44 933	<.1	10	4	< 2	4.4	214	.95	1.30
R7087	4 27	44.933	< 1	10	4.5	<.2	4.4	212	98	1.35
R7089	4 27	44 933	<.1	10	3	< 2	4.4	216	.94	1 29
R7090	4 27	44.653	≤ 1	10	3	<.2	4.5	215	.95	1 30
R7091	4.27	4.577	≤ 1	10	3	< 2	4.4	215	.95	1.30
R7092	4 27	44.933	<.1	10	3 5	≤ 2	4 4	214	.95	1 30
R7093	4.27	4 933	<.1	10	3 5	<.2	4.4	213	.95	1 30
R7095	4.27	44.552	<.1	10	4	≤ 2	4 5	214	.95	1 30
R7096	4 27	44 806	≤.1	10	4.5	≤ 2	4.4	218	92	1.27
R7097	4.27	44.933	<.1	10	5	≤ 2	4 4	218	.92	1 27
R7098	4 27	44 907	< 1	10	2.5	≤ 2	4 4	212	.98	1 35
R7099	4 27	44.806	≤ 1	10	3	<.2	4 4	212	98	1.35
R7116	4.27	45.847	<.1	10	1 5	<.2	4.4	216	.94	1 29

Table IV (Continued)

Test Results for Pilot Production Run Laser Rod Delivery

Rod Number	Rod Diameter (cm)	Rod Length (cm)	Surface Flatness (λ)	Parallelism (arc sec)	Perpendicularit (arc)	Strain (Fringes	Strain (Fringes/mm) X10⁻³	ifect (μs)	at Nd	Nd Conc (atom %)
R7116	4 27	45 364	≤.1	10	2 5	≤ 2	4.4	213	.97	1.33
R7118	4.27	45.339	<.1	10	2	≤ .2	4 4	212	.98	1 35
R7119	4.27	45.695	< 1	10	3.5	≤ .2	4 4	216	.94	1 29
R7120	4 27	45.847	≤.1	10	2	≤ .2	4.4	213	.97	1.33
R7120	4.27	45.923	≤ 1	10	3.5	< .2	4.4	215	.95	1.31
R7405	4 27	45 720	< 1	10	3	≤ 2	4 4	233	.77	1.06
R7406	4 27	45 720	< 1	10	3	≤ 2	4.4	226	.83	1 14
R7407	4.27	45 720	< 1	10	3	< 2	4.4	228	82	1 13
R7408	4.27	45 720	≤.1	10	5	< .2	4 4	234	.76	1 05
R7409	4 27	45 720	< 1	10	4	≤ 2	4 4	235	.74	1.02
R7410	4.27	45.720	≤ 1	10	4	< .2	4.4	229	81	1.11
R7411	4 27	45 720	< 1	10	4	≤ 2	4 4	236	73	1.00
R7412	4.27	45 720	≤.1	10	4	≤ .2	4.4	238	.70	.96
R7413	4 27	45 720	< 1	10	3	< .2	4.4	232	.78	1.07
R7414	4 27	45 720	≤.1	10	4	≤ 2	4.4	238	.70	96

Belvoir, Virginia. The delivery was made by personal carrier to Dr. A. Pinto on March 17, 1983. A group of six finished laser rods is shown in Figure 7 for reference.

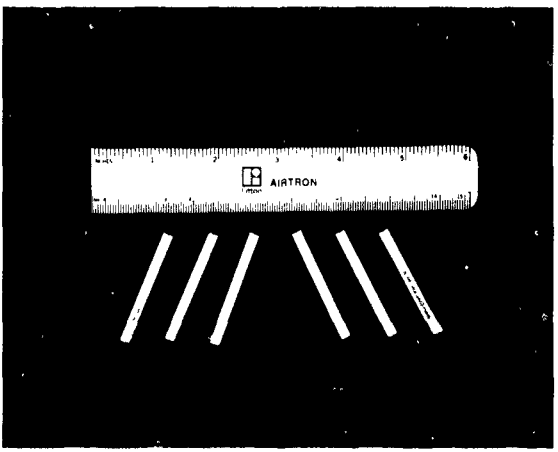


Figure 7. A group of six laser rods completely fabricated and coated. Rods were obtained from Boule 3122

APPENDIX VII

Test Equipment

Item	Requirements
Xenon flashlamp and power supply	Flashlamp duration of 100 μs
1.06 μm blocking filter	Less than 0.0001% transmission at 1.06μ
Detector	S-1 surface response
Oscilloscope - Tektronix Model 7704	Camera equipped with time base 100 μ sec/div.
Mitotoyu Calipers Model 505-637	Least count of 0.1 mm or better
Starrett Model 483 V-Anvil Micrometer	Least count of 0.01 mm or better
Nikon Model 6 Shadowgraph	Least count of 0.01 mm or better
Nikon Model 2100 Autocollimator	Resolution of 1 min of arc
Scratch & Dig Samples R.H. Beal Model 667 and viewing fixture Model 268	Per MIL-O-13830
Nikon Model 70702 binocular microscope and illuminator	10X magnification
Optical flats	Flat to $\lambda/10$
Fizeau Interferometer 632.8 nm laser source beam exapnsion optics viewing screen	Capable of positioning sample and measuring optical parallelism to 0.5 fringes (4 seconds)
Twyman-Green Interferometer Perkin Elmer Model 723	Capable of measuring wavefront distortion to $\lambda/10$
Tenny Model TH27 Temperature Humidity Chamber	Capable of attaining 95-100% humidity at 120°F ± 4°F

APPENDIX VII (Continued)

Item	Requirements
Cary Model 14 Spectrophotometer and reflectivity attachment	Capable of measuring reflectivities from 01.% to 100% at 1.06μ
He-Ne Gas Laser	1-3 mW output
Polarimeter	3 inch aperture and 8-10 inch length between polarizer and analyzer
Picker or General Electric X-ray Unit	Laue orientation camera for boules
Nd:YAG Laser Quantronix Model 114	Q-switch capability for coating damage tests
Extinction Ratio	High grade calcite polarizers
Coating Thickness Monitor	Optical technique for vacuum coater

APPENDIX VIII

Process Demonstration

In accordance with the contract provisions, a production capability demonstration was held at the Airtron plant on April 21, 1983. This full day demonstration reviewed the achievements and problems of the growth portion of the program. The agenda for the demonstration was centered around the following main items which were of prime interest to the U.S. Army Manufacturing Methods and Technology Engineering sponsors.

(a) The station arrangement and contruction was explained using a "cold" model. The preparation process was detailed for two inch growth.

(b) An actual two inch diameter growing boule was viewed in a "hot" crystal growth station.

(c) A processed boule from which the requisite number of laser rods had been fabricated was exhibited to demonstrate yield. Other grown two inch diameter boules of Nd:YAG were available from the program's process.

(d) The passive rod specifications were discussed in reference to program goals.

A list of attendees for this demonstration is given on the next page. All of the specific items shown at our demonstration are listed in Table VII of the following page. A similar Table VIII was prepared for some items not shown because of proprietary interests.

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AIRTRON

R. Lagno
R. Belt
R. Uhrin
G. Florio
R. Savas

Table VII

Specific Items Shown at Demonstration

Raw Material Component Oxides

4.5 x 4.5 Inch Iridium Crucibles and Lids

Nd:YAG [111] Seed Crystals

Ceramic Insulating Materials for Station

RF Coil around Crucible

Bell Jar Geometry

400 KHz RF Generator

Gas Arrangement and Flow System

Optical Control System

A Two Inch Diameter Crystal Growing in Melt

Drawings of Station Arrangement

Examples of Grown Boules and Difficulties

Station Set-up Process

Multirod Holder for 15 Rod Fabrication

Two Inch Diameter Boules and Sections

Actual Carcass from 60 Rod Boule

Fabricated (4 x 45)mm Laser Rods

Fabrication Process Charts

Quality Control Measurements Chart

Program Requirements and Delivery Charts

Table VIII

Items Not Shown at Demonstration

Proprietary Circuit Diagrams

Control System Interconnections

Proprietary Fabrication Processes

Quality Control Instrumentation

Company Developed Processes

Production Facilities Not Involved

Non-Contract Growth Data

APPENDIX IX

Identification of Personnel and Function

Dr. Roger Belt - General program responsibility for all growth, testing, reporting, and contract requirements.

Mr. Robert Uhrin - Project engineer for all growth, materials preparation, testing, and reporting.

Ms. Karen Grimes - Growth technician for all Nd:YAG boule preparations.

Mr. Steven Turner - Supervision of rod manufacturing and testing in process.

Mr. Robert Savas - Quality control engineer for optics and test procedures.

Mr. Douglas Cutter - Technician for quality control testing of Nd:YAG.

Mr. David Dentz - Chief engineer for all testing methods.

Mr. Gerald Florio - Manufacturing manager of all Nd:YAG production.

Appendix X

Nd:YAG Material and Rod Yields

Under our manufacturing program, details of crystal growth were examined to obtain boules of 50 mm diameter x 100mm long. The number of growth stations devoted to the experimental program was a maximum of two. Under these circumstances a limited number of growth runs were possible and a certain rod yield was obtained. In this section we would like to present a further discussion of yields as applied to our manufacturing process. It should be recalled that most crystal growth and processing data are statistical in nature and meaningful only when the sample is large; i.e. many runs are made, a lot more stations are in use, and hundreds of rods are fabricated. None of these could be achieved under a simple one-two year effort. Yet progress has been made in a manner similar to that in the past.

Our discussion begins with some elementary definitions of yields. The crystal growth yield is defined as the number of completed boules divided by the total number of run starts. This yield in production is about 85-90% because of a poor start, cracks, seed breakage, wrong melt, or other major difficulty related to instrumentation failure, power failure, crucible leak, or gas supply. Not much can be done with this yield other than to increase the reliability of all factors both human and physical.

The next important yield might be referred to as a rod yield from a boule. This is in reality a volume fraction of perfect boule and can be defined as the number of rods actually obtained divided by the rods which can be extracted if the boule were optically perfect. The rod definition is somewhat dependent on shape and diameter because smaller rods attain a better utilization of material. The (4.4 x 44.0)mm rod prepared under this program is a good example. At the start of the program, a rod yield of only 20-25% was obtained. Near the end of the program a rod of 60-75% was common. In a few cases we approached 90-95%. These were boules that were the best in all respects. A production reality for a large number of stations which operate over an extended period of time would be about 70%. At this writing not enough statistics have been generated to verify this yield explicitly.

The last yield under consideration is a fabrication yield. This yield is defined simply as the number of rods which are produced to the specification divided by the number which enter the processes. Here a rod may be rejected for breakage, length, wavefront distortion, bevel, parallelism, coating or other reason. Some of these rods may have reusable material or can be corrected by additional fabrication. The fabrication yield has remained consistently at 85-90% over many years of operating a facility which produces thousands of rods. Therefore the statistical basis is excellent and should not change much. About

80% of all rejected material can be used again and brought into correct specifications. For example, the length may be wrong, a coating defective, or some other minor item repaired and the rod will be useful.

The total yield is the product of the above individual yields and may run in the range of 45-60%. This does not appear to be high but the crystal growth process is most demanding. The melting point is near 1975°C for Nd:YAG, the growth rate is about 0.5mm/hr, constant temperature must be maintained to 10°C, and an optically perfect crystal is desired with a loss coefficient of 0.002 cm^{-1}. It should be recalled that a major power failure can affect up to 25-50 growth runs at a time. Fortunately this does not happen often at our facilities.

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1. B. Cockayne, Phil. Mag. 12, 943 (1965).

2. R. F. Belt, R.C. Puttbach, and D.A. Lepore, J. Crystal Growth 13/14, 268 (1972).

3. C. Belouet, J. Crystal Growth 15, 188 (1972).

4. R.F. Belt, R.C. Puttbach, J.R. Latore, and D. Dentz Contract No. DAAB 05-71-C-2611, January, 1973; Airtron, 200 East Hanover Ave., Morris Plains, N.J. 07950.

5. D. Dentz, Contract No. DAAB 07-77-C-0375, July, 1980; Airtron, 200 East Hanover Avenue, Morris Plains, N.J. 07950.

6. R. Uhrin and R. F. Belt, Manufacturing Methods and Technology Engineering for "Growth of Large Diameter Nd:YAG laser Crystals", Semiannual Progress Report, October 1, 1979 to April 1, 1980, Contract No. DAAB07-77-C-0375 Mod P0003, July, 1980.

7. Calculated with the aid of the General Electric Radiation Slide Rule GEN 15-C.

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